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MICROPOROUS FUMED-SILICA INSULATION AS A STANDARD REFERENCE MATERIAL OF THERMAL RESISTANCE AT HIGH TEMPERATURE

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**Microporous Fumed-Silica Insulation
as a Standard Reference Material
of Thermal Resistance**

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Measurements of apparent thermal conductivity of microporous fumed-silica insulation board, already certified as a Standard Reference Material (SRM) of thermal resistance, are reported here to extend the range of certification of this material to higher temperatures and lower pressures. Apparent thermal conductivities of five different pairs of specimens ranging in mean density from 300 to 348 kg/m³ were measured with a high-temperature guarded hot plate 25 cm in diameter. The measurements cover a range of mean specimen temperatures from 318 to 733 K (45 to 460°C), and of environmental air pressures from 26.7 to 83.5 kPa (200 to 626 Torr). Detailed analyses are given, along with intercomparisons of previously published data. Apparent thermal conductivity is correlated with temperature, pressure, and density; the correlation obtained represents all the data within a standard deviation of 0.68 percent. This microporous fumed silica (at an ambient pressure of 83 kPa and a density of 300 kg/m³) has an apparent thermal conductivity of 19.8 mW/(m·K) at 300 K and is suitable for use as an SRM of very low conductivity from 297 to 735 K (24 to 460°C). Adsorbed moisture within this material must be driven off by prolonged heating at 110°C before its conductivity is measured. Great care in handling this material is necessary because of its fragility.

Key words: apparent thermal conductivity; density; microporous fumed silica; insulation board; pressure; Standard Reference Material; temperature; thermal insulation; thermal resistance.

1. INTRODUCTION

The Office of Standard Reference Materials of the U.S. National Institute of Standards and Technology (NIST) establishes Standard Reference Materials (SRMs) to improve reliability in measurement of physical properties. Specifically, SRMs of thermal resistance are used by government, academic, and industrial laboratories to verify the correct operation of guarded hot plates (GHPs) and to calibrate instruments such as heat-flow meters. These instruments measure the rate of heat flow under given temperature differences through specimens of thermal insulation of known thickness. From such data, under steady conditions, one can obtain the thermal resistance (R-value) of the measured specimen as well as the apparent thermal conductivity of homogeneous material. SRMs are also used to indicate the accuracy of measurement of apparent conductivity of thermally conducting materials over a range of parameters such as temperature, bulk density, and environmental air pressure. The Properties of Solids Group within the Chemical Engineering Science Division of NIST in Boulder, Colorado (NIST-B), has participated for about 25 years in establishing many different SRMs, including SRMs of thermal resistance over a broad range of thermal conductivity, pressure, and temperature. The status of this effort has been summarized by Hust [1].

During the mid-1970's, the American Society for Testing and Materials (ASTM) recognized the great need for SRMs of thermal insulation. Consequently, a task-group was established under the auspices of ASTM subcommittee C16.30 on thermal measurements. Recommendations for establishing SRMs of thermal insulation were published in 1978 [2].

No SRM of thermal resistance for use at temperatures above 330 K is now available from the Office of Standard Reference Materials of NIST in Gaithersburg, Maryland (NIST-G), in spite of the very strong need for such SRMs. This work and another project now under way will add two SRMs of very different apparent thermal conductivity, both being suitable for use both at ambient and at higher temperatures, to fill this need. One of these SRMs, microporous fumed silica, is the subject of this report. The other, to be described in a separate publication [3], is a fibrous alumina-silica insulation board. It will be certified over the same range of temperature but will have an appreciably higher apparent thermal conductivity (55 mW/(m·K) at 323 K (50°C)) than the one described here. The two SRMs together will complement each other in range of apparent conductivity and in ease of handling.

Many insulation materials commonly used in practice contain, between their constituent particles and fibers, connected air spaces with characteristic sizes much larger than the mean free path of the air molecules occupying these spaces. The result is that the thermal conductivity of the material may follow rather closely the behavior of the thermal conductivity of air. Also, the contribution of the gas component to the thermal conductivity of

these materials is very nearly independent of air pressure, as is the total apparent thermal conductivity of the bulk material.

Unlike the apparent thermal conductivity of more commonly used insulation materials, that of the microporous fumed-silica insulation studied here depends markedly on the barometric pressure of the air surrounding the material and occupying its pores. This effect is due to the extremely small size and random close packing of the silica particles in this material. The random packing of these particles produces very small, irregularly shaped, connected pores having diameters, according to the manufacturer, of less than about 100 nm. This characteristic size is approximately the same as that of the mean free path of the occupying air molecules, which is about 66 nm [4] at standard atmospheric pressure and 288 K (15°C). As a result, (1) the contribution to the total apparent thermal conductivity from that of the solid component is greatly reduced because of the tortuosity of the conductive path through the very fine, solid particles; (2) the contribution of the thermal conductivity of the gas component to that of the bulk material is greatly reduced by the irregularity of the gas-conductive path through the very fine pores of the solid; (3) the thermal conductivity of the gas component depends significantly on pressure, as does the total apparent thermal conductivity of the bulk material.

The dependence of the apparent thermal conductivity of this microporous insulation material on ambient gas pressure has a practical consequence of great importance: the geographic elevation of the laboratory measuring the conductivity affects the value of apparent conductivity obtained. Therefore it is not enough just to know how the apparent conductivity depends on the temperature and density of the specimen. The absolute barometric pressure of the air filling the pores of this material must also be known in order to predict accurately the apparent thermal conductivity to be expected at the user's laboratory.

Here we report measurements of apparent thermal conductivity of five pairs of specimens of microporous fumed-silica insulation board. These pairs were obtained as a selected subset of specimens from the Heat Transfer Group of the Center for Building Technology at NIST-G, which has certified this material as an SRM at room temperature, based on measurements with their 1-meter GHP. This work at NIST-B has involved measurements of apparent thermal conductivity performed over a greater range of temperature and air pressure than was possible with the 1-m apparatus at NIST-G. Measured values were also correlated with the densities of the specimens.

The apparent thermal conductivity of this material lies in the lower range of values seen for typical insulation materials. Its conductivity is similar to that for closed-cell foam insulations of great practical importance in consumer applications such as in home-building and in home refrigerators and freezers. If care is taken in handling this very fragile material, it will potentially be of great use to those concerned with measuring apparent thermal

conductivity of foam insulations, with both guarded hot plates and heat-flow meters. Such instruments are used for quality control by manufacturers of thermal insulation and in developing new insulation products to conserve energy in domestic applications.

2. DESCRIPTION OF MICROPOROUS FUMED SILICA

This microporous fumed-silica insulation is produced as rigid boards of bonded silica-aerogel composite made of very fine particles of synthetic amorphous silica, opacifier, fine ceramic fibers, and a binder [5]. The size of the silica particles has been reported by the manufacturer to be approximately 10^{-8} m. The fine ceramic fibers increase the cohesiveness of the board, and the binder cements the surfaces of the particles and fibers together into a cellular structure by sintering when the boards are cured at high temperatures (900°C). Ilmenite ($\text{FeO} \cdot \text{TiO}_2$) is the opacifier added to reduce radiative heat transmission through the board, to make it suitable for use at high temperatures. The fractions of silica particles, opacifier, and ceramic fiber stand approximately in the ratios 60:35:5 by mass. All organic matter is burned out in curing.

Microporous fumed-silica insulation board is very fragile and must be handled with great care. The edges of the slabs are easily chipped during installation and removal from the measurement apparatus. Boards having an edge dimension longer than about 30 cm are easily damaged during handling and shipping unless great care is taken to support them over a whole face and to protect them from localized impacts. However, with proper cushioning of the specimens when they are packed for shipping, and with proper support of the broad faces when they are installed in an apparatus, a careful worker can probably measure them 20 or 30 times without significant degradation of their surfaces due to chipping or breaking. Great care will be necessary in handling these specimens.

Several evaluation lots of this insulation board were originally obtained by NIST-G [5]; the SRM candidate was selected from these. A second lot of the selected material became SRM 1449 in 1988 [6]. The specimens as received were about 2.5 cm thick and 60 cm square. After apparent thermal conductivity was measured in the 1-m GHP at NIST-G, selected pairs of specimens were cut into 30-cm squares and shipped to NIST-B. Specimens for which measurements of apparent thermal conductivity are reported here had mean densities of 301, 304, 315, 329 and 348 kg/m^3 (18.8 to 21.7 $\text{lb}_\text{m}/\text{ft}^3$).

3. APPARATUS AND MEASUREMENTS

The data for apparent thermal conductivity reported here were obtained with the high-temperature GHP described by Hust, Filla, Hurley, and Smith [7]. The outer diameter of this circular hot-plate stack, and of the specimens measured, is 255 mm (10 in); the diameter of the circular, concentric, metered main heater is 127 mm (5 in).

The guarded hot plate is designed to measure heat transfer by conduction, that is, by diffusive processes of heat transfer driven by temperature gradients. Because of the density and microporosity of this material, convective heat transfer is negligible at all temperatures and pressures used in this study. Analysis of our data in the following section suggests, however, that radiative heat transfer in this material is not negligible, but neither does it dominate. Near room temperature it contributes only a small amount in comparison to conductive heat transfer. At the upper end of the temperature range studied, thermal radiation becomes a greater fraction of the total heat transfer, but still contributes less than conduction does.

The insulation boards studied here are available in only one thickness, nominally one inch. Our apparatus is unable to measure two or more such thicknesses, and the fragility of this material makes machining specimens down to smaller thicknesses impractical. Therefore we cannot study the effect of specimen thickness and thereby begin unambiguously to separate the magnitude of the radiative contribution from the diffusive heat transfer. However it does appear that thermal conduction is the major contributor to the total heat transfer. Because conductive and radiative heat transfer are both present, interactively, in this insulation material, the term "thermal conductivity" alone is somewhat inaccurate and misleading as a descriptor of the heat-transfer behavior of this material over the temperature range studied. To call attention to this fact, the term "apparent thermal conductivity" will be used in the following text.

The microporous fumed-silica specimens were received as square slabs nominally 12 in (30.5 cm) on a side. The measurement apparatus requires disks 255 mm in diameter, so disks were cut from the square slabs by using a bandsaw set to a low cutting speed. The material is very easily cut and the disk was not damaged during the cutting. The annular material making up the rest of the square sometimes splits during the cutting, however. Because the material generates airborne dust during machining, prudence requires the use of a dust mask by the person cutting this material. Although the material from which the insulation is manufactured is not known to be toxic, the dust is very fine and is borne by stray air currents over distances of several meters.

We measured the thickness of each insulation board using a digital electronic caliper with a precision of 0.03 mm (0.001 in). Thickness was measured at eight locations equally spaced around the perimeter of the disk and the measurements were averaged. The standard deviation of the thickness measurements is 0.08 mm for a nominal specimen thickness of 25 mm (relative standard deviation of 0.3 percent).

Using a steel ruler having a precision of 0.25 mm, we measured the diameter of each specimen along four different directions, and averaged the results. The standard deviation of these four diameters was 0.8 mm for a nominal diameter of 255 mm (relative standard deviation of 0.3 percent).

The masses of the specimens were measured on a laboratory trip balance with a sensitivity of 0.1 g for a typical mass of about 400 g (relative imprecision of 0.03 percent).

The density was determined from these measurements with a precision of ± 0.5 percent. The mass of each specimen decreased by about 3 g from conditioning in an oven at 90°C before the thermal conductivity was measured. These changes in mass due to the rigors of conditioning and measurement amount to an uncertainty of about 0.8 percent in the density.

Time Required for Stability and Reproducibility

At the beginning of the measurement program the apparent conductivity was measured repeatedly to determine the minimum time required to obtain stable and reproducible data. Under automatic operation of the control system, conductivity at a mean specimen temperature of 60°C was measured at intervals averaging 7 h. The values obtained approached a final, steady value of 21.12 mW/(m·K) along an exponential-decay curve. The first value obtained was 22.16 mW/(m·K), at a time of 7 h into the series; the second value, 21.34 mW/(m·K), at 14 h, came within 1 percent of the final, stable value. The third value, at 21 h, was within 0.5 percent (the experimental reproducibility) of the stable value. Measurements performed at mean specimen temperatures of 440°C also required about 14 to 18 hours to lie within experimental reproducibility of the final, stable value. These observations led to the procedure described next.

After the fumed-silica specimens were installed in the guarded-hot-plate stack they were dried by being heated to the highest temperature of measurement (approximately 725 K, or 450°C) before any values of apparent thermal conductivity were obtained. This effectively removes almost all of the adsorbed moisture from the specimens and gives stable values of apparent thermal conductivity that are reproducible as long as the following measurements are performed at temperatures less than the highest temperature. No evidence of release of chemically bound water was detected.

For each measurement the temperature difference across a specimen was established at approximately one tenth of the absolute mean temperature in the specimen. Temperature differences smaller than this magnitude lead to systematic errors in apparent thermal conductivity [7]. At temperatures above approximately 800 K, the ceramic materials used in constructing the heater plates become electrically conductive; this leads to leakage currents coupling the thermocouples to the heater windings, and the alloys used in the thermocouples and resistance thermometers begin to degrade. For these reasons the highest temperature of the main heater plate was limited to 773 K (500°C), so the highest mean temperature used was about 733 K (460°C).

There is no evidence from the physical appearance of the specimens or in the apparent-conductivity data that phase changes occur in the material below 500°C, the highest temperature to which the specimens (that is, their sides adjacent to the hot main plate) were exposed during measurements. There does seem to be some adsorption of moisture by the specimens; this must be driven off before measurements become stable. We often found it necessary to repeat measurements two or more times at the highest mean temperature until the values obtained did not vary by more than the experimental reproducibility (0.5 percent for this apparatus). This required a period as long as 24 h to obtain a stable measurement. Then measurements of apparent conductivity below that temperature were stable and reproducible.

4. DEPENDENCE OF APPARENT CONDUCTIVITY ON TEMPERATURE AND DENSITY

The object of this work is to obtain the dependence of apparent thermal conductivity on three independent physical variables: temperature, density, and ambient air pressure. The complete apparent-conductivity function describing these relationships therefore occupies a four-dimensional space. Consequently it was much more convenient to study and to present the data in smaller subsets of lower numbers of variables. We shall therefore first treat apparent thermal conductivity as a function of temperature and density with ambient pressure constant. Then we shall study the apparent conductivity as a function of pressure, with the temperature and density held constant. These separate treatments will allow us to justify the mathematical form of the final correlation chosen to represent the dependence of apparent thermal conductivity on all three independent variables.

To determine the dependence on temperature and density, the apparent thermal conductivity of each pair of specimens was measured at mean temperatures from about 318 to 733 K (45 to 460°C) with air at ambient humidity and pressure (83 kPa, or 625 Torr in Boulder) in the environmental chamber surrounding the measurement stack. Absolute temperatures and temperature differences were stabilized and measured with a precision of 0.02 percent. The precision of measurements of apparent thermal conductivity with this apparatus has been estimated as 0.5 percent at 330 K, and 0.7 per-

cent at 720 K [7]. Measurements of apparent conductivity have a repeatability of about 0.75 percent. The bias of measurements obtained with this apparatus is estimated to be 1.0 percent for mean specimen temperatures near 320 K; this is based on recent measurements of a specimen of SRM 1450b performed over the range of temperatures from 315 to 370 K. The bias of measurements of apparent thermal conductivity is estimated to rise to no more than 2.5 percent at 725 K, as estimated from an analysis of propagation of errors based on sensitivity measurements performed on the GHP apparatus [7].

The five pairs of specimens of microporous fumed-silica insulation boards used in this study were identified by in-house codes as (a) B-08c and B-08d, (b) B-132 and B-133, (c) B-101 and B-102, (d) B-101 and B-104, and (e) B-073 and B-074. Because no member of a pair was measured individually, it will be simpler to refer to these pairs by the last digits of the individual identification codes of their two members. Thus the five pairs measured will be identified throughout this report respectively as (a) B8cd, (b) B23, (c) B12, (d) B14, and (e) B34.

(A) Expected Dependence of Conductivity on Temperature and Density

A simple phenomenological relation based on the following reasoning was chosen to represent the data. Any thermally insulating material in a gaseous atmosphere is composed of finely divided solid material permeated with the environmental gas. The thermal conductivities of gases and nonmetallic solids at ambient and higher temperatures have an approximately linear dependence on temperature. Black-body radiation depends on the fourth power of temperature of the emitting body, so heat transfer between two adjacent surfaces depends, to a very good approximation, on the product of the third power of the mean temperature with the temperature difference. The density of insulation material has two effects: transmission of thermal radiation decreases, and solid conduction increases, as the bulk density of the material increases. However, the increase in apparent thermal conductivity with density is less than proportional to the change in density. These considerations suggested the simple form of the dependence on density and temperature in the relation used here to represent the apparent-conductivity data.

(B) Experimental Results

In an environment of air at ambient humidity and at local ambient pressure of 82.5 to 83.4 kPa (619 to 626 Torr), 178 measurements of apparent thermal conductivity were made on five pairs of specimens with densities ranging from 301 to 348 kg/m³. Diameters of the specimens were 255 mm and thicknesses were 25.4 mm (1 in). Measurements covered the range of temperature from 318 to 733 K (45 to 460°C). The behaviors (apparent conductivity as a function of temperature) of all five pairs of specimens were qualitatively very similar.

The correlation of apparent thermal conductivity with temperature and density was represented by an expression of the form,

$$k = A_0 + A_1 \cdot D + A_2 \cdot T + A_3 \cdot T^3 / D, \quad (1)$$

where k is apparent thermal conductivity, T is absolute temperature, and D is density. The second term reflects the expected increase of apparent conductivity with bulk density of the material, and the inverse dependence on density in the fourth term reflects the expected decrease in radiative heat transport through the material as the density increases.

A correlation of this form was obtained using the thermal conductivity integral (TCI) method [8], with a weighted least-squares routine fitting both temperature and density simultaneously. The dependence on density was found from the statistical parameters of the fit to be small over this range of density, but significant.

The apparent thermal conductivity as a function of temperature had some curvature, but it was not strong. During these measurements, the temperature gradients imposed on the specimens were determined by the rule that the temperature difference should be about 10 percent of the absolute mean temperature. At all temperatures, such a temperature difference covers a small range over which the apparent-conductivity function is very nearly linear. Thus it would not be necessary to use the TCI method to fit the data for these specimens with acceptable accuracy. The TCI method should be used whenever measurements are made (1) with large differences in the boundary temperatures of the specimen and (2) with appreciable curvature in the apparent-conductivity function over the included range of temperature. Still, we use the TCI method as a matter of course, whether or not the data seem to have enough curvature to require the method. This ensures that the mathematics of our fitting procedure introduces no additional errors due to nonlinearity in the fitted function.

The standard deviation of the residuals between the curve given by eq (1) and the 178 data points was 0.68 percent. Standard deviations of the residuals for the individual pairs of specimens were:

- (a) B8cd, 45 residuals, 0.70 percent;
- (b) B23, 22 residuals, 0.35 percent;
- (c) B12, 42 residuals, 0.58 percent;
- (d) B14, 38 residuals, 0.30 percent;
- (e) B34, 31 residuals, 1.16 percent.

These values of standard deviations, which measure the goodness of the fit, compare very favorably with the value of the experimental reproducibility, 0.8 percent, for the GHP apparatus used. The dependence of apparent conductivity on temperature and density for this material is very well represented by a curve of the form of eq (1).

The densities of the specimens used in this work can be uncertain by about 1 percent due to adsorption and desorption of moisture during the measurements. The densities also vary slightly with mean specimen temperature, due to thermal expansion, but not enough to affect the dependence of apparent conductivity on density arrived at in this report. It was deduced from the numerical coefficients of the fit to eq (1), using values of temperature and density typical of the measurements reported here, that a change (or uncertainty) of 1 percent in density results in a change (inaccuracy) in values of apparent conductivity of no more than 0.2 percent. This is well within the experimental reproducibility. The uncertainty in temperature has a negligible impact on the accuracy of measurements of apparent thermal conductivity.

5. DEPENDENCE OF APPARENT CONDUCTIVITY ON AMBIENT AIR PRESSURE

(A) Constraints on the Ranges of Pressure Used in This Work

To study the effects of ambient air pressure, the apparent thermal conductivity was measured under several different conditions of air pressure and bulk density. We chose a range of air pressures that would also permit us to compare data taken at our local ambient pressure with complementary data obtained on the 1-m guarded hot plate at NIST-G. The elevation of our laboratory is approximately 1650 m above sea level, and the local ambient air pressure is typically about 84 kPa (625 Torr); the elevation of the laboratory at NIST-G is close to sea level, with a mean ambient air pressure of about 100 kPa (750 Torr).

Due to the large size and rectangular geometry of the environmental chamber containing the 1-m GHP at NIST-G, there are severe constraints on the allowed variation of air pressure within the chamber. A moderate difference between pressure within the chamber and the outside atmospheric pressure would damage its structure. As a result, data on the NIST Certificate for SRM 1449 (fumed-silica board) are tabulated only over the range from 97 to 102 kPa.

Within the environmental chamber surrounding the 25-cm GHP at NIST-B, air pressures at the specimens can be obtained over the range from local ambient, 84 kPa, down to pressures of less than about 1 Pa. However in this chamber an opposite constraint applies: air pressures greater than local ambient could be obtained only with difficulty and some danger. Due to the large horizontal cross section of the vacuum chamber, an internal lifting force of about 9 kN (1 ton) would be exerted on the roof on the chamber if it were pressurized to 101 kPa (1 atm). The energy stored within the large volume of pressurized air would not be negligible (about 10 kJ).

(B) Expected Dependence on Ambient Air Pressure

Over a wide range of environmental air pressure, the dependence of apparent thermal conductivity on pressure for commonly used insulation materials is qualitatively well understood. The curve for the dependence of apparent conductivity on pressure typically has a sigmoidal shape (figure 1; the values along both axes are arbitrary but representative). There is a region of constant, low conductivity at low pressure (A), a transitional region of rising conductivity at intermediate pressures (BCD), and a plateau of approximately constant, higher conductivity at higher pressures (E). The region connecting the rising transitional region to the high-pressure plateau (DE) is called the "knee." The low-pressure plateau of constant apparent conductivity is determined by radiative heat transfer and by conduction in the solid material, which are independent of pressure. The contribution from conduction in the gas within the pores of the bulk specimen determines the position and shape of the high-temperature plateau. There is no theory that completely describes the empirical pressure dependence depicted in figure 1 over the whole range of pressure; many different mathematical functions can be chosen to model the behavior approximately.

For the three pairs of boards used to study the dependence of apparent thermal conductivity on environmental air pressure, and at the four conditions of temperature and density used, the dependence of apparent conductivity on pressure was strong and clearly did not correspond to the high-pressure plateau. Instead, the conductivity-pressure relation for this material, for each condition of temperature and density, seemed to occupy only a narrow region of the transitional region below the knee (CD, figure 1), and appeared to be most simply described by a segment of a parabola.

(C) Measurements of Pressure

For the studies of dependence of apparent thermal conductivity on pressure discussed in this section, air pressure was measured with a Bourdon-tube pressure gauge with an imprecision of about 0.13 kPa (1 Torr); the relative imprecision is about 0.3 percent at the lowest pressures, and approaches 0.1 percent at the highest pressures used. The inaccuracy of the measurements of pressure is about 1 percent.

The ambient air pressures used in this work could vary by 0.5 percent (typical) to 1 percent (maximum observed) during the course of measurements, due to changes in barometric pressure. From the numerical coefficients of the fitted curve giving the dependence of apparent conductivity on pressure, using coefficients for pair B14 and values of temperature and pressure typical of the measurements reported here, we deduced that a change (or uncertainty) of one percent in pressure results in a change (inaccuracy) in values of apparent conductivity of about 0.3 percent. This is well within the experimental reproducibility of values of apparent thermal conductivity.

(D) Experimental Results

We measured the apparent thermal conductivity of three different pairs of specimens, representing three different bulk densities, over a range of air pressures of approximately 27 to 84 kPa (200 to 626 Torr). In particular, 20 values of apparent conductivity for specimen pair B14, with a mean density of about 301 kg/m³, were measured at a mean specimen temperature of 318 K. Nineteen values for pair B34, with a mean density of about 315 kg/m³, were measured at a mean specimen temperature of 473 K. Finally, pair B8cd, with a mean density of about 348 kg/m³, was measured both at 318 K (21 values) and at 473 K (22 values). A total of 82 measurements was made. Data for apparent thermal conductivity k as a function of ambient air pressure for all four pairs, individually as well as collectively, were well represented by a relation of the form

$$k(P) = B_0 + B_1 \cdot P + B_2 \cdot P^2, \quad (2)$$

where P is ambient air pressure surrounding the specimens. The sign of B_1 is positive, and that of B_2 is negative. All three coefficients are significant. The standard deviations of the residuals for the data for the four individual pairs of specimens ranged from 0.16 to 0.25 percent. Clearly a relation of the form of eq (2) describes well the dependence of apparent thermal conductivity on pressure.

6. DEPENDENCE OF APPARENT CONDUCTIVITY ON PRESSURE AND DENSITY

Specimen pairs B14 and B8cd were measured at 318 K. When the data for pressure dependence of apparent conductivity for these two pairs of two different densities were plotted together, the combined data for the two pairs were also well represented by a relation of the form of eq (2). The standard deviation of the 41 residuals between the data and the single fitted function was 0.66 percent. The dependence of apparent thermal conductivity on density for these two pairs is not large enough to be seen above the limits of experimental reproducibility.

Specimen pairs B34 and B8cd were measured at 473 K. When the data for the pressure dependence of apparent conductivity for these two pairs of different densities were plotted together, the combined data for the two pairs were also well represented by a relation of the form of eq (2). The standard deviation of the 41 residuals between the data and the single fitted function was only 0.37 percent. The dependence on density for these two pairs is also not large enough to be seen above the limits of experimental reproducibility.

The $k(P)$ curve for the two pairs of specimens at 318 K and the corresponding curve for the two pairs at 473 K were very similar in shape. This suggested that both sets of data might be accurately represented by a single function. The two sets of data were superimposed by the following technique.

The apparent-conductivity data for the two pairs at 318 K were adjusted by adding a constant to each conductivity datum. Then a curve of the form of eq (2) was fitted to the data for all four pairs, to all of which a temperature of 473 K effectively now applies. Trial and error found a constant additive term of 1.70 mW/(m·K) that gave the best fit between the curve and the data for the four pairs. For this fit the standard deviation of the 82 residuals between the curve and the data was only 0.50 percent.

The significance of this fact is that all four pressure relations for apparent thermal conductivity for two different temperatures and three different densities can be described by a function having a fixed shape. The position of the conductivity-pressure relation for one temperature is then determined from the position of the conductivity-pressure relation at another temperature by only the difference in apparent conductivities at the two different temperatures. In contrast to the situation with the measurements of apparent conductivity as a function of temperature, the data for apparent conductivity as a function of pressure is not sensitive to the density of the specimens used, so these data may be adjusted slightly as needed when combined with the conductivity-temperature data.

7. DEPENDENCE OF CONDUCTIVITY ON TEMPERATURE, DENSITY, AND PRESSURE

The relation between apparent thermal conductivity and pressure given by eq (2) can now be additively combined with the relation among apparent conductivity, density, and temperature given by eq (1) to obtain the correlation for apparent thermal conductivity of microporous fumed silica. As a function of all three independent variables, temperature, density and pressure, the apparent thermal conductivity k is

$$k(T,D,P) = 5.11 + 0.1478 \cdot P - 3.6996 \cdot 10^{-4} \cdot P^2 + 1.2064 \cdot 10^{-2} \cdot D \\ + 2.34402 \cdot 10^{-3} \cdot T + 5.8703 \cdot 10^{-6} \cdot T^3 / D, \quad (3)$$

where k is in units of $\text{mW}/(\text{m} \cdot \text{K})$, D is density in kg/m^3 , P is pressure in kPa , and T is temperature in K . The coefficients are determined by a total of 260 ($178 + 82$) measurements of apparent conductivity. The first three terms on the right side of this relation are consistent with eq (2) for the dependence of apparent conductivity on pressure; the first and last three terms are consistent with eq (1) for the dependence of apparent conductivity on temperature and density.

In eq (3) the constant term (5.11) was predicted from the combination of the fits to eqs (1) and (2) to be 5.18. However, while this value gave a nearly optimum fit for the conductivity-temperature data, it yielded residuals approaching 2 percent in magnitude for the conductivity-pressure data. The value of 5.11 was found by trial and error to give fits with approximately equal residuals, less than 2 percent, for both sets of data for apparent conductivity. This is not appreciably different from the predicted value.

Table 1(a-e) lists the 178 measurements of apparent thermal conductivity as a function of temperature and density, with pressure held constant at near 83 kPa . Data for each pair are listed on separate pages, with the density of the pair given at the top of the last column on each page. Also given are the values for apparent thermal conductivity calculated (K_{calc}) from the least-squares fitted curve, eq (3), as well as both absolute (K_{dev}) and relative (P_{dev}) deviations of the data from the curve. The apparent-conductivity data for each of the five individual pairs (at fixed density) are also plotted as a function of temperature in figures 2 through 11. Each odd-numbered figure illustrates the relative deviation, in percent, of the data from the calculated values, for the even-numbered preceding figure.

While calculating the fit, we allowed for the slight decrease in specimen density, from the nominal value listed in the table, accompanying increases in temperature. This variation in density is due to effects of thermal expansion of the thickness and area of the specimen. It slightly affects the values listed as k_{calc} . Likewise the values for the measured apparent conductivity, k_{dat} , were also corrected for the effects of thermal expansion.

The collective value of the relative standard deviation of the residuals between eq (3) and all five sets of temperature data combined with all four sets of pressure data was 0.68 percent. This is only slightly greater than the separate values for apparent conductivity as a function of temperature or for apparent conductivity as a function of pressure. The extremal deviations of the data from eq (3) were within ± 2.2 percent. We consider the fact that one dependent variable is fitted here to three independent variables, with the experimental imprecision being 0.8 percent. In this light, for a relation in three variables as simple as eq (6), the standard deviation seems to be acceptably small; the extremal deviations are a little larger than desired but are judged not inconsistent with the combined effects of the standard deviation (0.68 percent), the total number of data fitted (260), and the value of experimental imprecision (0.8 percent).

For all five pairs of this microporous fumed-silica insulation, the apparent thermal conductivity is approximately 20 mW/(m·K) at 300 K and 83 kPa. This is substantially lower than the thermal conductivity of dry air, which is approximately 25.6 mW/(m·K) at 300 K and 101.3 kPa [9]. Although the lower ambient air pressure is responsible for part of the reduced conductivity, it does not explain the total difference. The microporous structure of the material seems to reduce the apparent conductivity of this insulation to a value below that of free air, despite the presence of conduction through the solid matrix of silica particles, in parallel with (and additive to) conduction through the permeating gas (air). This is probably due to the greatly reduced mean free path of the air molecules within the very small pores of the solid structure.

The curvature of the solid line in each even-numbered figure, 2 through 10, is correlated with the T^3/D term in eq (1), which, being nonlinear, grows larger relative to the other two terms with increasing temperature. This T^3/D term is related to the contribution of radiative heat transfer to the total heat transfer through the material. At 297 K the magnitude of this term is about 0.5 mW/(m·K) out of a total apparent conductivity of about 20 mW/(m·K), or about 2.5 percent of the total. At 700 K the magnitude of the T^3 term is about 6.7 mW/(m·K) out of a total of 27 mW/(m·K), or 25 percent of the total. Thus in this range of temperature, the radiative contribution to the total heat transfer is becoming appreciable, but conductive heat transfer still dominates.

To illustrate the goodness of fit between the data for apparent conductivity as a function of pressure and eq (3), plots of apparent thermal conductivity are provided as a function of pressure for each of the pairs and conditions from which the pressure dependence was derived. Figures 12 and 14 give the apparent conductivity data for pairs B14 and B8cd at fixed temperatures of 318 K, and figures 13 and 15 are the corresponding deviation plots. Similar information for pairs B34 and B8cd at fixed temperatures of 473 K are given in figures 16 through 19. The data for the individual pairs are all listed in tables 2 through 5.

The correlations of apparent thermal conductivity with temperature for all five pairs compose a family of five curves, each for a given density. It is useful to illustrate the explicit dependence of the $k(T,D)$ relation on density, giving a family of curves at constant temperature. However, it was inconvenient to use the original data for apparent conductivity as a function of density, because the actual data cannot be grouped precisely into coherent isotherms. To illustrate the density dependence, eq (1) was used to calculate the apparent conductivity arbitrarily at temperatures of 300 to 800 K, at intervals of 100 K, and at the five densities describing the actual five pairs of measured specimens. The points corresponding to these conditions are shown in figure 20. Deviation plots are not given here because the points used are calculated; however the points have an experimental basis through the data upon which eq (1) is based.

For this material the rate of change of apparent thermal conductivity with density, holding temperature constant, is given mathematically by the first partial derivative of the right side of eq (3) with respect to the variable D , holding terms in temperature and pressure constant. Carrying out this operation gives

$$\begin{aligned}\partial k / \partial D &= A_1 + A_3 \cdot T^3 / D^2 \\ &= 0.012064 - 5.8703 \cdot 10^{-6} \cdot T^3 / D^2, \quad (4)\end{aligned}$$

so that the variation of apparent conductivity with change in density depends both on the temperature and on the density itself. In particular, as T increases with D fixed, the magnitude of the second (negative) term grows, so the slope decreases with increasing temperature (at constant density). As D increases with T fixed, the magnitude of the second term is reduced, so the slope increases with increasing density (at constant temperature).

We evaluate this expression for temperatures of (a) 300 K, (b) 600 K, and (c) 800 K, and compare the results with the curves of figure 20. At these three temperatures, and at a density of 300 kg/m³ (the lowest value measured here), the rates of change of apparent conductivity (in units of mW/(m·K)) with density (in kg/m³) are respectively (a) $10.3 \cdot 10^{-3}$, (b) $-2.0 \cdot 10^{-3}$, and (c) $-21.3 \cdot 10^{-3}$. At a density of 348 kg/m³ (the highest value measured here), these rates of change are respectively (a) $10.7 \cdot 10^{-3}$, (b) $1.6 \cdot 10^{-3}$, and (c) $-12.8 \cdot 10^{-3}$. From these calculated values, or equivalently, from figure 20, it is seen that (a) the curve for 300 K has a small, approximately constant positive slope; (b) the curve for 600 K has a slope near zero; and (c) the curve for 800 K has a negative slope, of magnitude greater than that for 300 K, and decreasing in magnitude as density increases. The range of density covered here is not great enough for figure 20 to show unambiguously that the curvature is upward for all five curves, that is, that the slope increases as density increases. But this effect is shown by the numerical values cited here for each temperature.

Returning to eq (4) for the rate of change of apparent conductivity with density at constant pressure, we compute the rate of change of apparent conductivity with density for a temperature of 318 K and a mean density of 324 kg/m³ (mean of 300 and 348 kg/m³, the densities of pairs B14 and B8cd). The value obtained is $10.3 \cdot 10^{-3}$ for apparent conductivity in mW/(m·K) and density in kg/m³. For a temperature of 348 K and a mean density of 332 kg/m³ (mean of 315 and 348 kg/m³, the densities of pairs B34 and B8cd), the rate of change of apparent conductivity with density is $9.8 \cdot 10^{-3}$. The difference between the two values is not greatly significant for our purpose here; the average is about $10 \cdot 10^{-3}$ mW/(m·K).

The slopes of the six different lines in figure 20 have a physical origin. For a given mean temperature within a thermal insulation, there is an optimum density that minimizes the apparent thermal conductivity. At densities lower than this optimum value, apparent conductivity rises because radiative heat transfer is not efficiently blocked; at densities higher than the optimum, conductivity rises due to the increased fraction of material that is solid, leading to greater conductive heat transfer. Thus the curves for 300 and 400 K show that the optimum density is less than 295 kg/m³, while the curves for 700 and 800 K indicate that the optimum density is greater than 350 kg/m³. For temperatures of 500 to 600 K the range of densities included on the graph is near the optimum value. The small numerical values of the nonzero slopes show that, for all curves from 300 to 750 K, the minimum in the relation between apparent conductivity and density is very broad. Therefore the optimum density is imprecisely located, but on the other hand the apparent thermal conductivity is not very sensitive to departures from the optimum density.

Because $k(P)$ as given by eq (2) depends quadratically on P , the rate of change of k with pressure is pressure-dependent. The coefficient B_1 in eq (3) was positive, and B_2 negative, so the slope of k versus P is positive but decreases as pressure increases. The rate of change of apparent conductivity with pressure, or slope of the $k(P)$ function plotted in figure 12 (or 14, 16 or 18), is given by the mathematical first derivative of $k(P)$. The derivative of the right side of eq (2) takes the form

$$dk/dP = B_1 + 2 \cdot B_2 \cdot P. \quad (5)$$

For pair B14, as an example, this relation takes the form

$$dk/dP = 0.17436 - 1.12246 \cdot 10^{-3} \cdot P, \quad (5')$$

for k in $mW/(m \cdot K)$ and P in kPa . At sea level, where P_{s1} is about 101 kPa , the rate of change of apparent conductivity with pressure, dk/dP from eq (5'), takes the value 0.0606 for pair B14 (with a density of 300 kg/m^3). At the elevation of our laboratory at Boulder, where the pressure is $P_B = 83.5$ kPa , the rate of change of conductivity with pressure is 0.0806. But because these slopes are different (k depends nonlinearly on P), neither of them is valid for extrapolating values of k obtained at one ambient pressure to a different range of pressure. Rather, the $k(P)$ relation itself, eq (3), should be used to estimate the apparent conductivity to be expected at a given ambient pressure.

8. COMPARISONS

The microporous fumed-silica insulation board studied here comes from the same lot that has already been measured [6] and certified [10] by Ebberts, Somers, and Zarr at NIST-G, for use as an SRM of low apparent thermal conductivity. All but three published measurements were performed at a mean specimen temperature of 297 K (24°C) and at a pressure of 101.33 kPa (760 Torr), on specimens having densities ranging from 310 to 333 kg/m³.

The certificate [10] for SRM 1449 (fumed silica) furnishes both tabular data, and an algebraic formula (coefficients as given here have been modified to give apparent conductivity for heat flow in milliwatts),

$$k_G(297\text{ K}) = 6.9943 + 0.021375 \cdot D + 0.070723 \cdot P, \quad (6)$$

relating apparent thermal conductivity k_G in mW/(m·K), density D in kg/m³, and ambient air pressure P in kPa (subscript G on k denoting "as determined at NIST-G"). This correlation is valid only for a temperature of 297 K. The specimens on which eq (6) is based covered a somewhat smaller range of density (300 to 330 kg/m³) and were measured over a considerably smaller range of pressure (97 to 102 kPa) than the measurements reported here; the certification data and accompanying table imply that these limits define the range of applicability of eq (6).

The coefficient of density, $21.375 \cdot 10^{-3}$, in eq (6) gives the rate of change of apparent thermal conductivity with density at constant temperature (297 K) and pressure. It was obtained for a fit of apparent conductivity to both density and pressure as independent variables. This coefficient is about twice the value we found, $10 \cdot 10^{-3}$. The paper describing the certification work [6] gave a value of $19.92 \cdot 10^{-3}$ for the density coefficient in a simplified correlation of apparent conductivity with density alone. A still earlier paper [5] listed a value of $30.76 \cdot 10^{-3}$ for this coefficient, for a separate lot of material from the same manufacturer, before the effect of moisture content was known. Thus there is evidence that the value for the density coefficient is sensitive to moisture content and lot of manufacture, as well as to the number of variables handled in fitting the correlation. The differences between these numbers also depend on the effects of combined experimental imprecision in developing an empirical correlation among several independent parameters (apparent thermal conductivity with temperature, pressure, and density).

The correlation of apparent conductivity with density obtained here may be compared with a set of 15 data points obtained at NIST-G for specimens at 297 K and having densities ranging from 304 to 325 kg/m³. This set is published in reference [6] as open squares in a figure, and also in a table. We show these same data, replotted here in figure 20 as open squares. The data plotted in our figure 20 from our correlation for specimens of five different densities and at 300 K are also replotted in figure 21, as open circles. The five circles lie well below the 15 squares for data from NIST-G. However, we must remember that these data (circles) apply to specimens measured at 83.2 kPa. Using either eq (2) or eq (3), we find that, for fixed density and temperature, the effect of increasing ambient air pressure from 83.2 kPa (NIST-B) to 101.3 kPa (NIST-G) is to increase the apparent conductivity by an additive contribution of 1.43 mW/(m·K). The five open circles in figure 21 have therefore been corrected for the influence of ambient air pressure by being moved upward 1.43 mW/(m·K) and replotted as "plus" signs. Now the disagreement is only about 0.5 mW/(m·K). The corrected NIST-B data deviate from the NIST-G data by a relative difference of about +2 percent.

To compare the correlation of apparent conductivity with temperature obtained here with results obtained at NIST-G, we choose one specimen, B23, with a density (328 kg/m³), very nearly equal to that of a specimen described in reference [6] which had a density of 330 kg/m³. Comparison may be made only after correcting our data for the effect of ambient air pressure on the apparent conductivity. Figure 22 shows the original data obtained here for pair B23, previously shown in figure 3. The single set of data from NIST-G for dependence of apparent conductivity on temperature was obtained at three different temperatures. The measurements listed in Table 4 of reference [6] are

Mean Temperature K	Apparent thermal conductivity mW/(m·K)
283	21.06
297	21.31
311	21.51

These 6 data from NIST-G are plotted in figure 22 as a filled square, triangle, and diamond, along with data obtained here for pair B23 (open triangles). The filled symbols lie well above the open triangles. The same correction must be applied to the data to correct for the effect of ambient air pressure. The same term used previously with the density correlation, 1.43 mW/(m·K), was added to the NIST-B data, obtaining the numbers shown in figure 22 as plus signs. The agreement between the data for NIST-G and NIST-B is now very good. The filled triangle and diamond agree with the corrected data within experimental scatter; the filled square may be about 1 percent lower than the trend line.

At the elevation of the NIST-B laboratory in Boulder, Colorado, 1650 m (5400 ft), the mean atmospheric pressure is approximately 83.2 kPa, with normal fluctuations, due to local weather, of ± 0.9 kPa. Table 2 gives the dependence of apparent conductivity on varying ambient air pressure for pair B14, at approximately 318 K. From the first row we find, for an air pressure of 83.4 kPa (626 Torr), near the maximum pressure used, that $k_{\text{dat}} = 20.07$ mW/(m·K), and $k_{\text{calc}} = 19.87$. By extrapolating eq (3) to a condition of mean atmospheric pressure at sea level, or 101.3 kPa, the apparent thermal conductivity at $T = 297$ K is estimated to be $k_{\text{s1}} = 21.11$ mW/(m·K). This value is higher than the value $k_{\text{calc}} = 19.87$ for our elevation of 1650 m, by 1.24 mW/(m·K). We found earlier that the curve for dependence of conductivity on pressure has the same shape, within experimental error, independent of density and temperature.

The Certificate for SRM 1449 states that values of measured apparent conductivity (on the NIST-G GHP) should agree with Certificate values within 1.5 percent, where apparent conductivity is measured at 297 K and within the range of pressure from 97 to 102 kPa. The NIST-B GHP has a claimed accuracy of 1 percent. The combined inaccuracies of measurements by the two laboratories could be as great as 2.5 percent, especially since values from each laboratory are estimated here at conditions somewhat outside the range of validity of the correlation for that lab. The agreement between the two laboratories is judged acceptable, considering the combined experimental uncertainties.

Another similar insulation material of low apparent conductivity, made of microporous fumed silica and having a nominal density of about 310 kg/m³, was studied by Tye [12]. He measured specimens from four different lots and found very small variations in conductivity between the lots. His results reproduced those of the manufacturer within 4 percent up to temperatures higher than 450°C. For these reasons he recommended in 1970 that it be considered as a possible SRM. Its apparent conductivity ranged from 26.5 to 48.5 mW/(m·K) at temperatures from 25 to 650°C. These values are somewhat higher than the apparent conductivities found here for this material made by a different manufacturer, but are in fair agreement with them. It is known that materials of similar densities but from different manufacturers will yield different apparent thermal conductivities [5].

9. REFERENCES

- [1] Hust, J.G. "Status of Thermal Conductivity Standard Reference Materials at the National Bureau of Standards", **THERMAL CONDUCTIVITY 18: Proceedings of the 18th International Conference on Thermal Conductivity**, Ashworth, T. and Smith, David R., Eds.; pp. 327-338 (Plenum, New York, 1985).
- [2] ASTM Subcommittee C16.30. "Reference Materials for Insulation Measurement Comparisons", **Thermal Transmission Measurements of Insulation**, ASTM STP 660, Tye, R.P., Ed.; pp. 7-29 (ASTM, Philadelphia, 1978).
- [3] Smith, David R., "Fibrous Alumina-Silica Insulation Board as a Standard Reference Material of Thermal Resistance", to be published.
- [4] F.W. Sears and G.L. Salinger, **Thermodynamics**, p. 290 (Addison-Wesley, Reading, Massachusetts, 1975).
- [5] Rennex, B.G., Somers, T.A., Faison, T.K., and Zarr, R.R., "Evaluation of Fumed-Silica Insulation for a Thermal Conductivity Standard Reference Material", **Insulation Materials, Testing and Applications**, ASTM STP 1030 (ASTM, Philadelphia, 1989).
- [6] Zarr, R.R., Somers, T.A. and Ebberts, D.F. "Room-Temperature Thermal Conductivity of Fumed-Silica Insulation for a Standard Reference Material", NBSIR 88-3847, (U.S. National Institute of Standards and Technology, June 1988).
- [7] Hust, J.G., Filla, B.J., Hurley, J.A. and Smith, D.R. "An Automated High Temperature Guarded-Hot-Plate Apparatus for Measuring Thermal Conductivity of Insulation Between 300 and 750 K", NBSIR 88-3089 (U.S. National Institute of Standards and Technology, May 1988).
- [8] Hust, J.G., and Lankford, A.B. "Comments on the Measurement of Thermal Conductivity and Presentation of a Thermal Conductivity Integral Method", *Int. J. of Thermophysics*, 3/1, 67-76 (1982).
- [9] F. Kreith and W. Black, **Basic Heat Transfer**, Appendix G, p. 520 (Harper & Row, New York, 1980).
- [10] "National Institute of Standards and Technology Certificate, Standard Reference Material 1449, Thermal Resistance - Fumed Silica Board", NIST, Gaithersburg, MD, 1989.
- [11] Tye, R.P. "The Thermal Conductivity of MINK-2000 Thermal Insulation in Different Environments to High Temperatures", *Proc. of 9th Conference on Thermal Conductivity*, CONF-691002, pp. 341-351 (U.S. Atomic Energy Commission, Div. of Technical Information, March 1970).

Table 1. Apparent thermal conductivity of five pairs of micro-porous fumed-silica insulation boards with mean densities of (a) 348, (b) 328, (c) 305, (d) 300, and (e) 315 kg/m³, at mean temperatures ranging from 318 to 723 K (45 to 450°C). One hundred and seventy-eight measurements were performed in air at ambient humidity and at pressures of approximately 83 kPa (625 torr), using the high-temperature guarded hot plate at the National Institute of Standards and Technology in Boulder, Colorado. Temperature differences through the specimens were chosen to be about 10 percent of the value of the mean temperature within the specimens. The bias in values of K_{meas} is estimated to be 1.5 percent at 330 K, and 2.5 percent at 720 K. The column labeled K_{calc} gives values of apparent thermal conductivity calculated from eq (3). The collective standard deviation of the 178 residuals between the data (K_{dat}) and the fitted curve is 0.68 percent. Absolute (K_{dev}) and relative (P_{dev}) deviations compare measured (K_{dat}) to calculated (K_{calc}) values of thermal conductivity.

Table 1a. Apparent thermal conductivity of pair B8cd as a function of temperature.

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
059	653.181	592.922	623.052	82.51	24.76	24.53	0.23	0.94	348
060	708.283	638.063	673.173	82.51	25.86	25.73	0.13	0.50	
061	708.098	638.001	673.050	82.51	25.86	25.73	0.13	0.50	
062	708.211	638.084	673.148	82.51	25.98	25.73	0.25	0.96	
065	708.134	638.194	673.164	82.51	26.06	25.73	0.33	1.27	
066	748.370	678.234	713.302	82.51	27.20	26.82	0.38	1.41	
067	748.087	678.163	713.125	82.51	27.20	26.81	0.39	1.46	
070	748.109	678.070	713.090	82.51	27.18	26.81	0.37	1.39	
071	707.944	638.027	672.986	82.51	25.91	25.73	0.18	0.69	
072	652.692	593.131	622.912	82.51	24.72	24.53	0.19	0.78	
073	602.810	543.148	572.979	82.51	23.60	23.50	0.10	0.43	
077	548.148	498.143	523.146	82.51	22.87	22.62	0.25	1.10	
078	522.940	473.073	498.007	82.51	22.46	22.22	0.24	1.08	
079	498.049	448.121	473.085	82.51	21.95	21.87	0.09	0.40	
080	468.026	428.111	448.069	82.51	21.54	21.54	0.00	0.02	
081	443.079	403.121	423.100	82.51	21.16	21.24	-0.08	-0.37	
082	418.084	378.122	398.103	82.51	20.91	20.96	-0.06	-0.28	
083	338.120	308.127	323.124	82.51	20.30	20.29	0.01	0.04	
084	338.153	308.170	323.162	82.51	20.33	20.29	0.03	0.15	
085	368.278	328.207	348.243	82.51	20.55	20.50	0.05	0.26	
086	393.210	353.153	373.182	82.51	20.88	20.72	0.16	0.79	
090	333.235	303.190	318.213	83.04	20.19	20.30	-0.11	-0.53	
091	333.153	303.151	318.152	83.04	20.20	20.30	-0.11	-0.53	
096	363.151	333.149	348.150	83.04	20.57	20.54	0.03	0.16	
097	366.176	329.588	347.882	83.04	20.64	20.54	0.10	0.49	
098	363.143	333.154	348.149	83.04	20.52	20.54	-0.02	-0.09	
099	338.295	308.220	323.258	83.04	20.34	20.34	0.00	0.00	
100	341.215	305.226	323.221	83.04	20.40	20.34	0.06	0.31	
101	338.157	308.163	323.160	83.04	20.35	20.34	0.01	0.05	
102	368.159	328.087	348.123	83.04	20.67	20.54	0.13	0.63	
103	371.177	325.177	348.177	83.04	20.62	20.54	0.08	0.39	
104	368.126	328.068	348.097	83.04	20.55	20.54	0.00	0.02	
105	393.239	353.141	373.190	83.04	20.90	20.76	0.14	0.66	
106	396.167	350.155	373.161	83.04	20.77	20.77	0.01	0.04	
109	418.170	378.151	398.161	82.64	21.12	20.98	0.15	0.70	
110	420.167	376.171	398.169	82.64	21.10	20.98	0.12	0.59	
111	418.122	378.129	398.126	82.64	21.13	20.98	0.16	0.74	
112	448.272	398.141	423.207	82.64	21.43	21.25	0.18	0.84	
113	451.177	395.150	423.164	82.64	21.38	21.25	0.13	0.61	
114	448.135	398.146	423.141	82.64	21.36	21.25	0.11	0.52	
116	473.168	423.111	448.140	82.64	21.72	21.55	0.17	0.78	
117	476.190	420.145	448.168	82.64	21.68	21.55	0.12	0.58	
118	473.107	423.128	448.118	82.64	21.68	21.55	0.13	0.59	
119	498.244	448.134	473.189	82.64	22.07	21.88	0.19	0.87	
120	503.243	443.235	473.239	82.64	22.00	21.88	0.12	0.55	

STANDARD DEVIATION = 0.70

Table 1b. Apparent thermal conductivity of pair B23 as a function of temperature.

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
256	758.120	688.183	723.152	83.17	27.18	27.29	-0.11	-0.40	328
258	733.087	663.257	698.172	83.17	26.48	26.55	-0.07	-0.26	
259	710.971	635.124	673.048	83.17	25.74	25.86	-0.12	-0.45	
260	708.132	638.140	673.136	83.17	25.77	25.86	-0.08	-0.33	
261	683.061	613.263	648.162	83.17	25.10	25.21	-0.11	-0.44	
262	656.124	590.154	623.139	83.17	24.53	24.60	-0.08	-0.32	
263	653.132	593.178	623.155	83.17	24.54	24.60	-0.06	-0.24	
264	628.057	568.103	598.080	83.17	23.94	24.04	-0.10	-0.42	
265	606.044	540.117	573.081	83.17	23.44	23.52	-0.08	-0.35	
266	603.123	543.136	573.130	83.17	23.50	23.52	-0.02	-0.09	
267	578.067	518.144	548.106	83.17	22.97	23.04	-0.07	-0.29	
269	548.108	498.119	523.114	83.17	22.55	22.59	-0.04	-0.17	
271	526.172	470.002	498.087	83.17	22.29	22.19	0.10	0.44	
272	501.077	445.054	473.066	83.17	21.89	21.81	0.07	0.34	
273	498.122	448.041	473.082	83.17	21.92	21.81	0.11	0.52	
274	473.262	423.156	448.209	83.17	21.61	21.47	0.14	0.65	
275	446.115	400.147	423.131	83.17	21.20	21.16	0.04	0.20	
276	443.132	403.126	423.129	83.17	21.26	21.15	0.10	0.49	
279	398.173	348.188	373.181	83.17	20.65	20.62	0.04	0.17	
281	368.143	328.155	348.149	83.17	20.37	20.38	-0.02	-0.09	
283	356.152	310.757	333.455	83.17	20.25	20.26	0.00	-0.02	
284	353.153	313.157	333.155	83.17	20.25	20.25	0.00	0.00	

Standard Deviation = 0.35

Table 1c. Apparent thermal conductivity of pair B12 as a function of temperature.

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
302	498.183	448.071	473.127	83.17	21.87	21.67	0.20	0.94	305
303	498.274	448.069	473.172	83.17	21.88	21.67	0.21	0.98	
305	548.184	508.095	528.140	83.17	22.65	22.60	0.05	0.24	
306	603.323	542.847	573.085	83.17	23.47	23.50	-0.03	-0.12	
310	758.098	688.084	723.091	83.17	27.37	27.54	-0.17	-0.63	
311	728.113	658.150	693.132	83.17	26.41	26.60	-0.18	-0.69	
312	730.173	655.069	692.621	83.17	26.56	26.58	-0.02	-0.07	
313	728.119	638.098	683.109	83.17	26.23	26.31	-0.07	-0.28	
314	708.163	638.201	673.182	83.17	26.06	26.01	0.06	0.22	
315	733.259	663.169	698.214	83.17	26.87	26.75	0.12	0.46	
316	758.324	688.206	723.265	83.17	27.56	27.55	0.01	0.04	
317	760.159	686.055	723.107	83.17	27.57	27.55	0.02	0.09	
318	751.047	675.142	713.095	83.17	27.15	27.22	-0.07	-0.26	
319	748.105	678.189	713.147	83.17	27.23	27.22	0.01	0.05	
320	735.004	661.088	698.046	83.17	26.76	26.75	0.01	0.04	
321	733.113	663.125	698.119	83.17	26.81	26.75	0.06	0.24	
322	710.142	636.144	673.143	83.17	26.09	26.01	0.09	0.33	
323	708.130	638.178	673.154	83.17	26.09	26.01	0.08	0.33	
324	683.069	613.537	648.303	83.17	25.41	25.31	0.09	0.36	
325	655.996	590.102	623.049	83.17	24.71	24.66	0.05	0.22	
326	653.108	593.162	623.135	83.17	24.82	24.66	0.17	0.67	
327	633.075	573.113	603.094	83.17	24.19	24.17	0.01	0.06	
328	608.024	538.134	573.079	83.17	23.57	23.50	0.07	0.28	
329	606.123	540.129	573.126	83.17	23.60	23.50	0.10	0.41	
330	603.156	543.146	573.151	83.17	23.63	23.50	0.13	0.57	
331	578.048	518.115	548.082	83.17	23.00	22.98	0.02	0.09	
332	551.044	495.115	523.080	83.17	22.56	22.51	0.05	0.24	
333	548.190	498.153	523.172	83.17	22.68	22.51	0.17	0.77	
334	523.088	473.143	498.116	83.17	22.12	22.07	0.05	0.22	
335	501.054	445.128	473.091	83.17	21.80	21.67	0.13	0.58	
336	498.121	448.132	473.127	83.17	21.86	21.67	0.19	0.88	
337	473.095	423.152	448.124	83.17	21.43	21.30	0.12	0.58	
338	445.083	400.185	422.634	83.17	21.07	20.96	0.11	0.52	
339	443.291	403.166	423.229	83.17	21.24	20.97	0.27	1.27	
340	423.085	373.136	398.111	83.17	20.84	20.67	0.17	0.82	
341	393.049	353.137	373.093	83.17	20.47	20.39	0.07	0.37	
342	376.093	330.125	353.109	83.17	20.39	20.19	0.20	0.98	
343	373.126	333.136	353.131	83.17	20.35	20.19	0.16	0.80	
344	353.113	313.181	333.147	83.17	20.20	20.01	0.19	0.96	
345	348.113	318.139	333.126	83.17	20.13	20.01	0.12	0.62	
346	343.134	313.165	328.150	83.17	20.11	19.97	0.15	0.74	
347	338.143	308.157	323.150	83.17	20.09	19.92	0.17	0.83	

Standard Deviation = 0.58

Table 1d. Apparent thermal conductivity of pair B14 as a function of temperature.

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
401	443.256	403.184	423.220	83.57	21.00	20.98	0.02	0.09	300
402	441.168	405.093	423.131	83.57	20.93	20.98	-0.05	-0.25	
403	443.168	403.099	423.134	83.57	20.95	20.98	-0.03	-0.12	
404	445.166	401.159	423.163	83.57	20.95	20.98	-0.03	-0.14	
405	603.281	543.078	573.180	83.57	23.60	23.53	0.07	0.29	
406	708.294	637.800	673.047	83.57	25.96	26.06	-0.10	-0.40	
409	758.146	688.102	723.124	83.57	27.71	27.62	0.09	0.33	
410	737.959	658.086	698.023	83.57	26.78	26.82	-0.04	-0.14	
411	735.060	661.128	698.094	83.57	26.86	26.82	0.04	0.14	
412	733.157	663.205	698.181	83.57	26.86	26.82	0.04	0.16	
413	713.031	633.139	673.085	83.57	26.06	26.07	-0.01	-0.04	
414	711.104	635.156	673.130	83.57	26.07	26.07	0.00	0.02	
415	708.141	638.187	673.164	83.57	26.09	26.06	0.02	0.08	
416	706.160	640.261	673.211	83.57	26.06	26.06	0.00	-0.01	
417	681.099	615.150	648.125	83.71	25.36	25.37	-0.01	-0.03	
418	653.024	593.135	623.080	83.71	24.65	24.71	-0.06	-0.26	
419	653.150	593.229	623.190	83.71	24.63	24.72	-0.09	-0.37	
420	653.151	593.201	623.176	83.71	24.69	24.72	-0.03	-0.12	
421	628.057	568.134	598.096	83.71	24.00	24.11	-0.11	-0.45	
422	605.207	541.133	573.170	83.71	23.52	23.55	-0.02	-0.10	
423	603.127	543.159	573.143	83.71	23.52	23.55	-0.02	-0.10	
424	601.125	545.221	573.173	83.71	23.53	23.54	-0.01	-0.05	
425	578.078	518.148	548.113	83.71	22.96	23.02	-0.06	-0.27	
426	550.107	496.166	523.137	83.71	22.43	22.54	-0.11	-0.50	
427	548.151	498.170	523.161	83.71	22.51	22.54	-0.03	-0.15	
428	546.152	500.169	523.161	83.71	22.55	22.54	0.01	0.04	
429	523.120	473.165	498.143	83.71	21.99	22.10	-0.11	-0.50	
430	500.134	446.159	473.147	83.71	21.65	21.70	-0.05	-0.22	
431	498.134	448.130	473.132	83.71	21.65	21.70	-0.05	-0.21	
432	496.097	450.108	473.103	83.71	21.62	21.70	-0.07	-0.35	
433	391.242	355.150	373.196	83.71	20.38	20.41	-0.03	-0.17	
434	393.187	353.153	373.170	83.71	20.31	20.41	-0.10	-0.48	
435	395.141	351.120	373.131	83.71	20.36	20.41	-0.05	-0.23	
436	418.205	378.168	398.187	83.71	20.69	20.69	0.01	0.03	
438	341.115	306.888	324.002	83.71	19.79	19.95	-0.16	-0.79	
439	339.181	307.182	323.182	83.71	19.81	19.94	-0.13	-0.66	
440	340.177	306.394	323.286	83.71	19.90	19.94	-0.04	-0.21	
441	337.172	305.412	321.292	83.71	19.89	19.92	-0.04	-0.18	

Standard Deviation = 0.30

Table 1e. Apparent thermal conductivity of pair B34 as a function of temperature.

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
503	758.300	687.931	723.116	83.04	27.76	27.40	0.35	1.30	315
504	773.111	692.596	732.854	83.04	28.07	27.72	0.36	1.28	
505	771.047	694.546	732.797	83.04	28.10	27.71	0.38	1.37	
506	768.085	697.949	733.017	83.04	28.13	27.72	0.41	1.47	
507	706.264	540.143	623.204	83.04	24.91	24.68	0.22	0.90	
508	708.213	638.115	673.164	83.04	26.21	25.91	0.29	1.14	
509	710.143	636.099	673.121	83.04	26.19	25.91	0.27	1.06	
510	678.082	618.152	648.117	83.04	25.42	25.24	0.18	0.73	
512	653.166	593.211	623.189	83.04	24.89	24.61	0.28	1.13	
513	651.106	595.169	623.138	83.04	24.89	24.61	0.28	1.13	
514	628.121	568.216	598.169	83.04	24.25	24.03	0.22	0.90	
515	605.155	541.203	573.179	83.04	23.62	23.50	0.13	0.54	
516	498.252	448.158	473.205	83.44	21.81	21.76	0.05	0.23	
517	578.376	518.169	548.273	83.04	23.24	23.00	0.24	1.05	
518	548.098	498.124	523.111	83.04	22.60	22.53	0.07	0.31	
519	523.086	473.116	498.101	83.04	22.17	22.11	0.06	0.28	
539	471.239	425.202	448.221	83.04	21.50	21.37	0.14	0.64	
541	333.203	304.985	319.094	83.04	20.16	20.00	0.16	0.81	
542	338.107	308.111	323.109	83.04	20.18	20.03	0.15	0.75	
543	340.083	306.837	323.460	83.04	20.17	20.03	0.14	0.70	
544	343.273	313.273	328.273	83.04	20.16	20.07	0.09	0.44	
545	346.222	310.223	328.223	83.04	20.36	20.07	0.29	1.44	
546	353.195	313.179	333.187	83.04	20.41	20.12	0.30	1.48	
547	355.243	311.665	333.454	83.04	20.45	20.12	0.34	1.67	
548	367.983	328.002	347.993	83.04	20.57	20.24	0.33	1.61	
549	366.118	330.051	348.085	83.04	20.69	20.25	0.45	2.21	
551	402.406	363.342	382.874	83.04	20.83	20.58	0.25	1.19	
552	404.422	360.877	382.650	83.04	20.84	20.58	0.26	1.25	
553	416.088	380.081	398.085	83.04	20.90	20.75	0.15	0.72	
556	418.235	378.193	398.214	83.04	21.12	20.75	0.37	1.77	
557	446.232	402.098	424.165	83.44	21.30	21.08	0.22	1.05	

STANDARD DEVIATION = 1.16

Table 2. Apparent thermal conductivity of pair B14 of microporous fumed-silica insulation boards measured at a mean specimen temperature of 318 K (45°C), and over the range of pressure from 40 to 83 kPa (300 to 626 torr), with the high-temperature guarded hot plate. This pair has a mean density of 301 kg/m³ (19 lbm/ft³). Apparent conductivity was measured in air at ambient humidity. Temperature differences through the specimens were chosen to be about 10 percent of the value of the mean absolute temperature within the specimens. Absolute and relative deviations compare measured apparent conductivity to values of Kcalc computed from eq (3).

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mw/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
442	331.151	305.119	318.135	83.44	20.07	19.87	0.20	0.99	301
443	333.152	304.224	318.688	83.44	20.05	19.88	0.17	0.85	
444	334.130	304.259	319.195	83.44	19.97	19.88	0.09	0.45	
445	331.143	305.150	318.147	83.04	19.91	19.84	0.07	0.37	
446	331.095	305.076	318.086	79.97	19.63	19.57	0.06	0.29	
447	332.209	304.294	318.252	73.31	19.10	18.96	0.13	0.71	
448	333.133	304.090	318.612	73.31	19.05	18.97	0.09	0.46	
449	334.140	304.205	319.173	73.31	19.09	18.97	0.12	0.62	
450	335.229	304.469	319.849	66.64	18.45	18.34	0.11	0.60	
451	332.142	304.149	318.146	59.98	17.81	17.65	0.16	0.91	
452	331.164	305.155	318.160	53.32	17.08	16.95	0.14	0.81	
453	332.147	304.151	318.149	53.32	17.06	16.95	0.11	0.67	
454	331.167	305.178	318.173	53.32	17.02	16.95	0.07	0.42	
455	332.141	304.142	318.142	46.65	16.28	16.21	0.07	0.42	
456	331.121	305.109	318.115	46.65	16.25	16.21	0.04	0.24	
457	332.185	304.187	318.186	39.99	15.44	15.44	0.01	0.05	
458	333.123	303.818	318.471	39.99	15.38	15.44	-0.06	-0.40	
459	332.142	304.138	318.140	39.99	15.40	15.44	-0.04	-0.26	
460	331.152	305.167	318.160	59.98	17.77	17.65	0.12	0.66	
461	332.125	304.120	318.123	59.98	17.80	17.65	0.15	0.85	

Standard Deviation = 0.62

Table 3. Apparent thermal conductivity of pair B8cd of microporous fumed-silica insulation boards measured at a mean specimen temperature of 318 K (45°C), and over the range of pressure from 27 to 80 kPa (200 to 600 torr), with the high-temperature guarded hot plate. This pair has a mean density of 348 kg/m³ (22 lbm/ft³). Apparent conductivity was measured in air at ambient humidity. Absolute and relative deviations compare measured apparent conductivity to values of Kcalc computed from eq (3).

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
149	333.199	303.114	318.157	47.05	16.50	16.73	-0.23	-1.39	348
150	335.160	301.180	318.170	39.99	15.76	15.91	-0.16	-0.98	
151	333.145	303.135	318.140	39.99	15.71	15.91	-0.20	-1.25	
152	335.153	301.179	318.166	39.99	15.75	15.91	-0.17	-1.04	
153	333.140	303.139	318.140	39.99	15.67	15.91	-0.24	-1.52	
154	335.147	301.160	318.154	39.99	15.74	15.91	-0.18	-1.11	
155	333.139	303.122	318.131	39.99	15.68	15.91	-0.23	-1.43	
156	335.144	301.188	318.166	53.32	17.29	17.42	-0.13	-0.76	
157	333.141	303.139	318.140	33.32	14.82	15.11	-0.29	-1.89	
158	335.159	301.181	318.170	26.66	13.97	14.27	-0.30	-2.13	
159	333.127	303.098	318.113	66.64	18.56	18.80	-0.24	-1.30	
160	335.154	301.181	318.168	66.64	18.61	18.80	-0.19	-1.00	
161	333.141	303.091	318.116	66.64	18.56	18.80	-0.24	-1.26	
162	335.155	301.163	318.159	66.11	18.64	18.75	-0.11	-0.59	
163	333.139	303.094	318.117	73.04	19.24	19.42	-0.17	-0.88	
164	335.158	301.179	318.169	59.98	18.00	18.13	-0.13	-0.72	
165	333.139	303.087	318.113	59.98	17.93	18.13	-0.20	-1.12	
166	335.159	301.158	318.159	79.97	19.86	20.05	-0.19	-0.96	
167	333.199	303.115	318.157	79.97	19.88	20.05	-0.17	-0.84	
168	335.161	301.177	318.169	79.97	19.93	20.05	-0.11	-0.57	
169	333.147	303.138	318.143	79.97	19.88	20.05	-0.17	-0.83	

Standard Deviation = 1.22

Table 4. Apparent thermal conductivity of pair B34 of microporous fumed-silica insulation boards measured at a mean specimen temperature of 473 K (200°C), and over the range of pressure from 40 to 83 kPa (300 to 626 torr), with the high-temperature guarded hot plate. This pair has a mean density of 315 kg/m³ (19.7 lbm/ft³). Apparent conductivity was measured in air at ambient humidity. Absolute and relative deviations compare measured apparent conductivity to values of Kcalc computed from eq (3).

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
516	498.252	448.158	473.205	83.44	21.81	21.76	0.05	0.23	315
520	501.086	445.137	473.112	83.44	21.75	21.76	-0.01	-0.03	
521	498.116	448.086	473.101	73.31	20.91	20.85	0.07	0.32	
522	496.134	450.131	473.133	73.31	20.95	20.85	0.10	0.50	
523	498.177	448.172	473.175	66.64	20.28	20.21	0.07	0.36	
524	500.161	446.139	473.150	66.64	20.31	20.21	0.10	0.50	
526	498.294	448.226	473.260	59.98	19.63	19.54	0.09	0.46	
527	501.048	445.019	473.034	59.98	19.62	19.53	0.08	0.42	
528	498.088	448.029	473.059	53.32	18.90	18.83	0.07	0.38	
529	495.101	451.108	473.105	53.32	18.91	18.83	0.09	0.46	
530	498.139	448.137	473.138	53.32	18.95	18.83	0.12	0.62	
531	500.070	446.066	473.068	53.32	18.82	18.83	0.00	-0.02	
532	498.136	448.133	473.135	46.65	18.10	18.09	0.01	0.05	
533	496.192	450.190	473.191	39.99	17.34	17.32	0.03	0.14	
534	498.037	448.035	473.036	39.99	17.32	17.32	0.00	0.00	
535	500.204	446.339	473.272	59.98	19.58	19.54	0.04	0.20	
536	497.984	448.002	472.993	79.97	21.62	21.45	0.17	0.80	
537	496.110	450.121	473.116	79.97	21.59	21.45	0.14	0.64	
538	498.395	448.268	473.332	79.97	21.53	21.46	0.07	0.33	
539	498.395	448.268	473.332	37.99	17.07	17.08	-0.01	-0.06	

STANDARD DEVIATION = 0.41

Table 5. Apparent thermal conductivity of pair B8cd of microporous fumed-silica insulation boards measured at a mean specimen temperature of 473 K (200°C), and over the range of pressure from 27 to 84 kPa (200 to 630 torr), with the high-temperature guarded hot plate. This pair has a mean density of 348 kg/m³ (22 lbm/ft³). Apparent conductivity was measured in air at ambient humidity. Absolute and relative deviations compare measured apparent conductivity to values of Kcalc computed from eq (3).

Run No.	Thigh (K)	Tlow (K)	Tmean (K)	Press (kPa)	Kdat	Kcalc mW/(m.K)	Kdev	Pdev (Pct)	Density (kg/m ³)
123	498.156	447.973	473.065	39.99	17.36	17.52	-0.16	-0.94	348
124	498.179	448.010	473.095	26.66	15.73	15.88	-0.16	-0.98	
125	501.171	445.119	473.145	26.66	15.70	15.88	-0.18	-1.14	
126	498.143	448.149	473.146	26.66	15.70	15.88	-0.18	-1.14	
127	498.146	448.155	473.151	26.66	15.71	15.88	-0.18	-1.11	
128	498.143	448.152	473.148	26.66	15.69	15.88	-0.20	-1.23	
129	498.145	448.099	473.122	26.66	15.71	15.88	-0.17	-1.09	
130	498.147	448.142	473.145	33.32	16.53	16.72	-0.19	-1.14	
131	498.140	448.125	473.133	46.65	18.06	18.30	-0.24	-1.29	
132	498.142	448.144	473.143	53.32	18.76	19.03	-0.28	-1.45	
133	501.169	445.186	473.178	53.32	18.80	19.04	-0.23	-1.22	
134	498.137	448.126	473.132	53.32	18.73	19.03	-0.31	-1.61	
135	501.156	445.154	473.155	59.98	19.44	19.74	-0.30	-1.52	
137	501.181	445.161	473.171	66.64	20.24	20.41	-0.18	-0.87	
138	498.133	448.123	473.128	73.31	20.77	21.05	-0.29	-1.36	
139	501.166	445.192	473.179	73.31	20.76	21.05	-0.29	-1.40	
140	498.142	448.140	473.141	73.31	20.76	21.05	-0.30	-1.41	
141	501.167	445.170	473.169	79.97	21.36	21.66	-0.30	-1.40	
142	498.135	448.139	473.137	79.97	21.33	21.66	-0.33	-1.53	
143	501.162	445.179	473.171	79.57	21.33	21.63	-0.29	-1.36	
144	498.131	448.132	473.132	79.57	21.32	21.62	-0.30	-1.39	
145	501.170	445.129	473.150	83.71	21.65	21.99	-0.34	-1.55	

Standard Deviation = 1.32

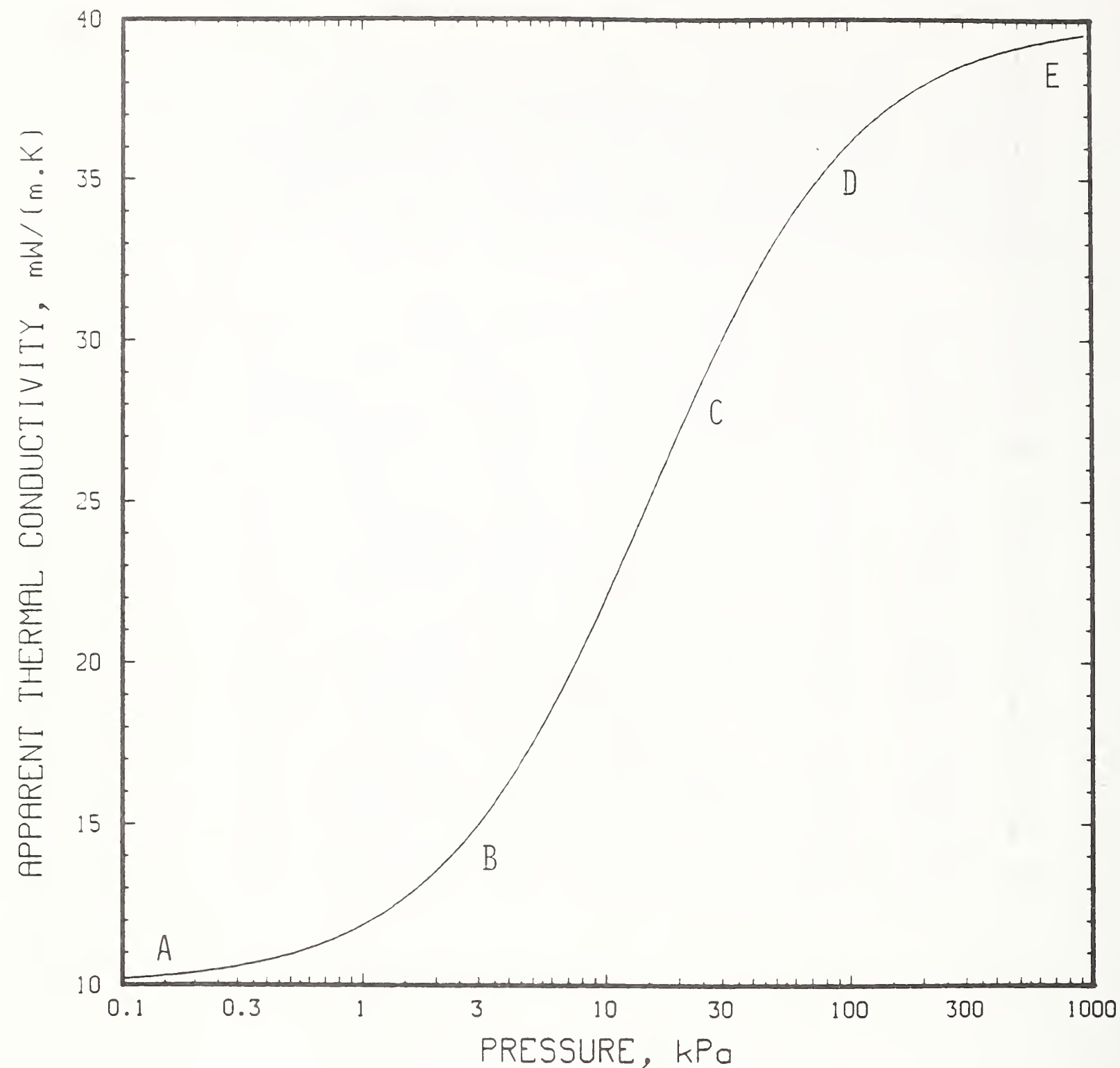


Figure 1. Typical dependence of apparent thermal conductivity of microporous fumed-silica insulation on ambient air pressure. No theory exists to completely predict the sigmoidal shape modelling the dependence. The values along both axes are arbitrary but representative. In this figure A is the region of constant, low conductivity at low pressure; B-C-D is the transitional region of rising conductivity at intermediate pressures; E is the beginning of the plateau of approximately constant, higher conductivity at higher pressures. The region connecting the rising transitional region to the high-pressure plateau (D-E) is the "knee". The low-pressure plateau of constant apparent conductivity is determined by radiative heat transfer and by conduction in the solid material, which are independent of pressure.

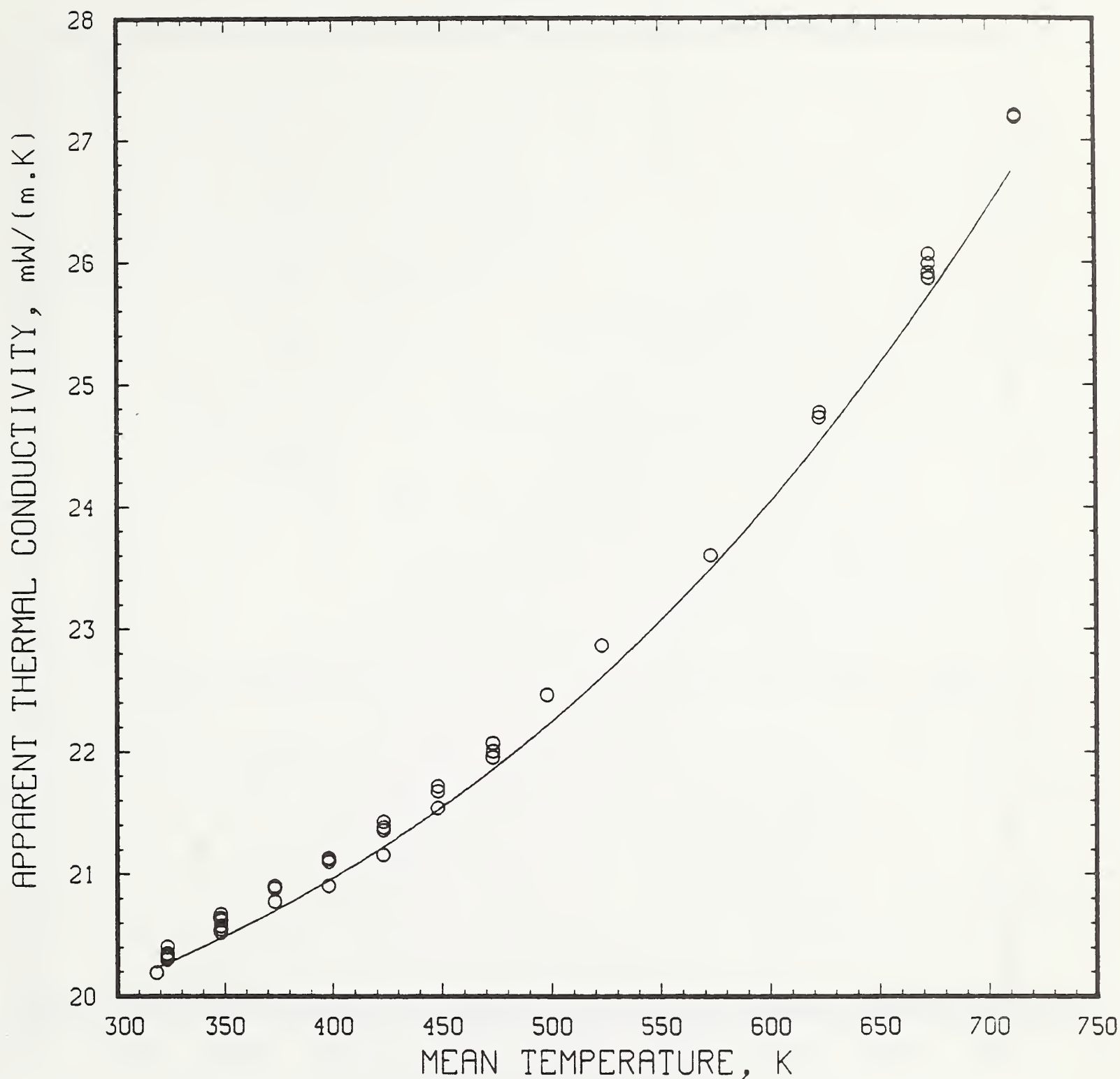


Figure 2. Apparent thermal conductivity of pair B8cd of micro-porous fumed-silica insulation at mean specimen temperatures ranging from 318 to 713 K (45 to 440°C), with the NIST-B high-temperature guarded hot plate. This pair has a mean density of 348 kg/m³ (22 lbm/ft³). Conductivity was measured in air at ambient humidity and at a pressure of 83 kPa. The measurement bias in values of K_{meas} is estimated to be 1.0 percent at 320 K, and 2.5 percent at 725 K. The solid curve represents values of K_{calc} obtained using eq (3).

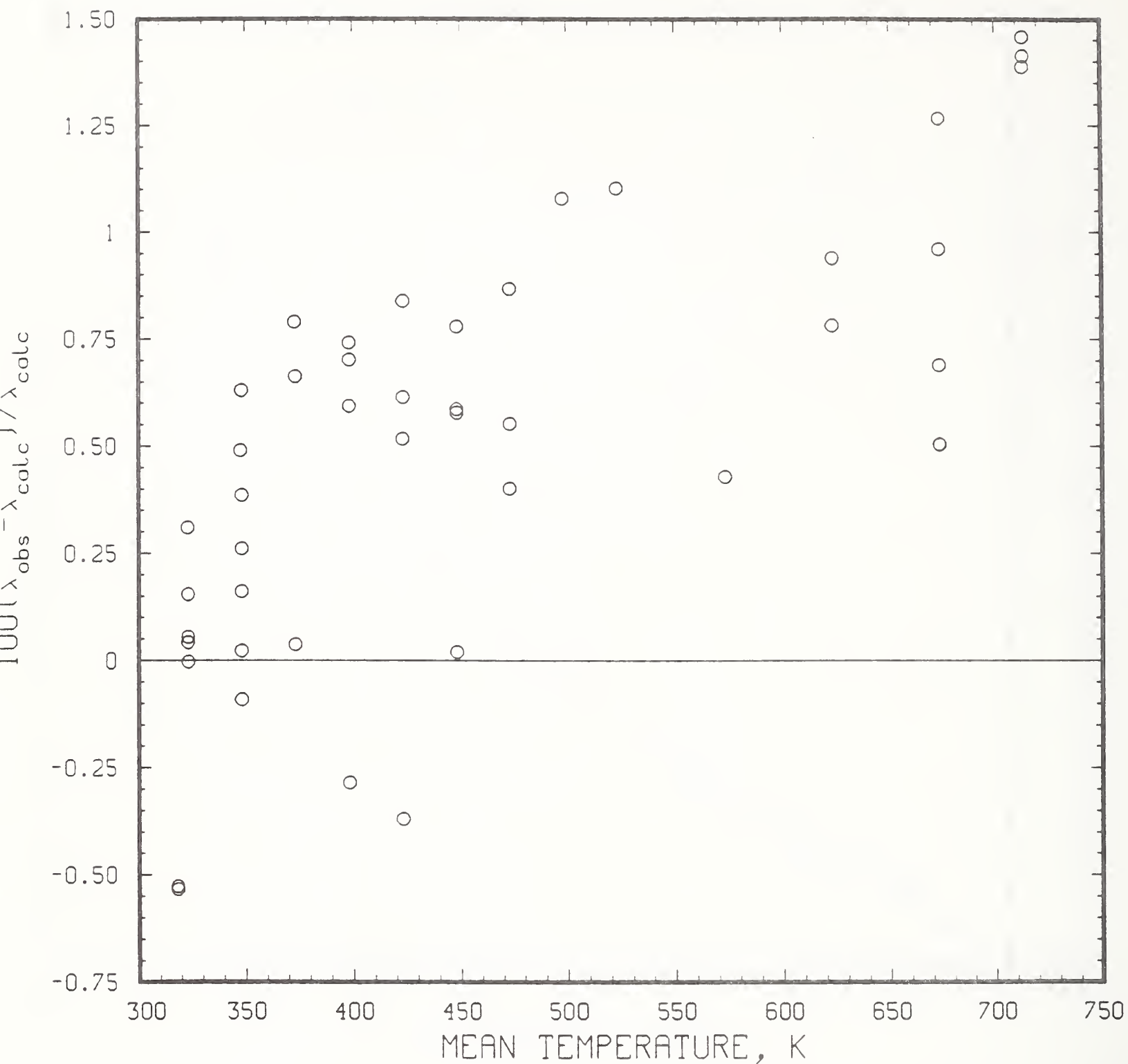


Figure 3. Relative (percent) deviations of thermal conductivity of fumed-silica insulation pair B8cd from values calculated with eq (3). The standard deviation of the 45 residuals between the points and the curve is 0.70 percent.

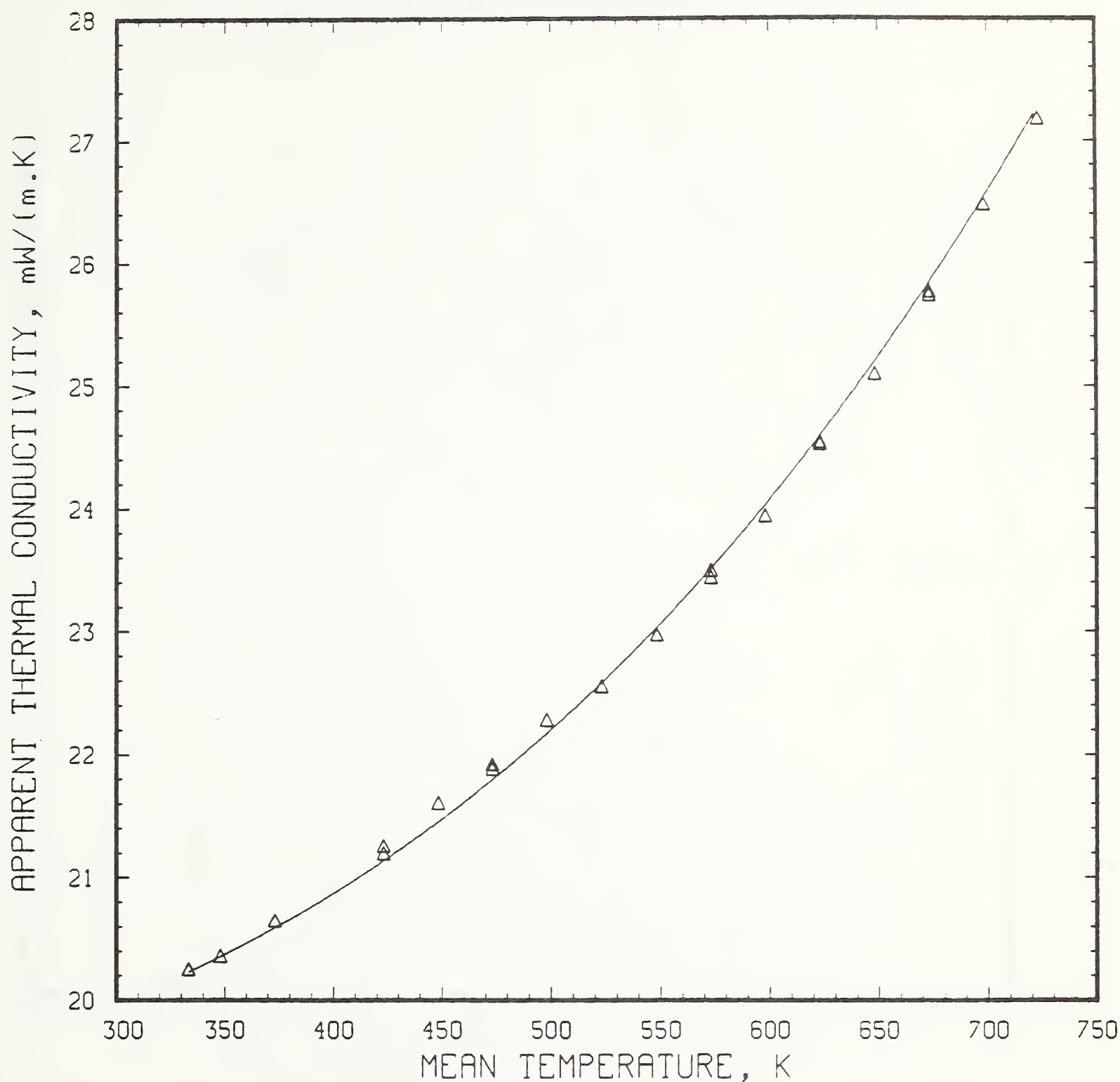


Figure 4. Apparent thermal conductivity of pair B23 of micro-porous fumed-silica insulation at mean specimen temperatures ranging from 333 to 723 K (60 to 450°C). This pair has a mean density of 328 kg/m³ (20 lbm/ft³). Conductivity was measured in air at ambient humidity and at a pressure of 83 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

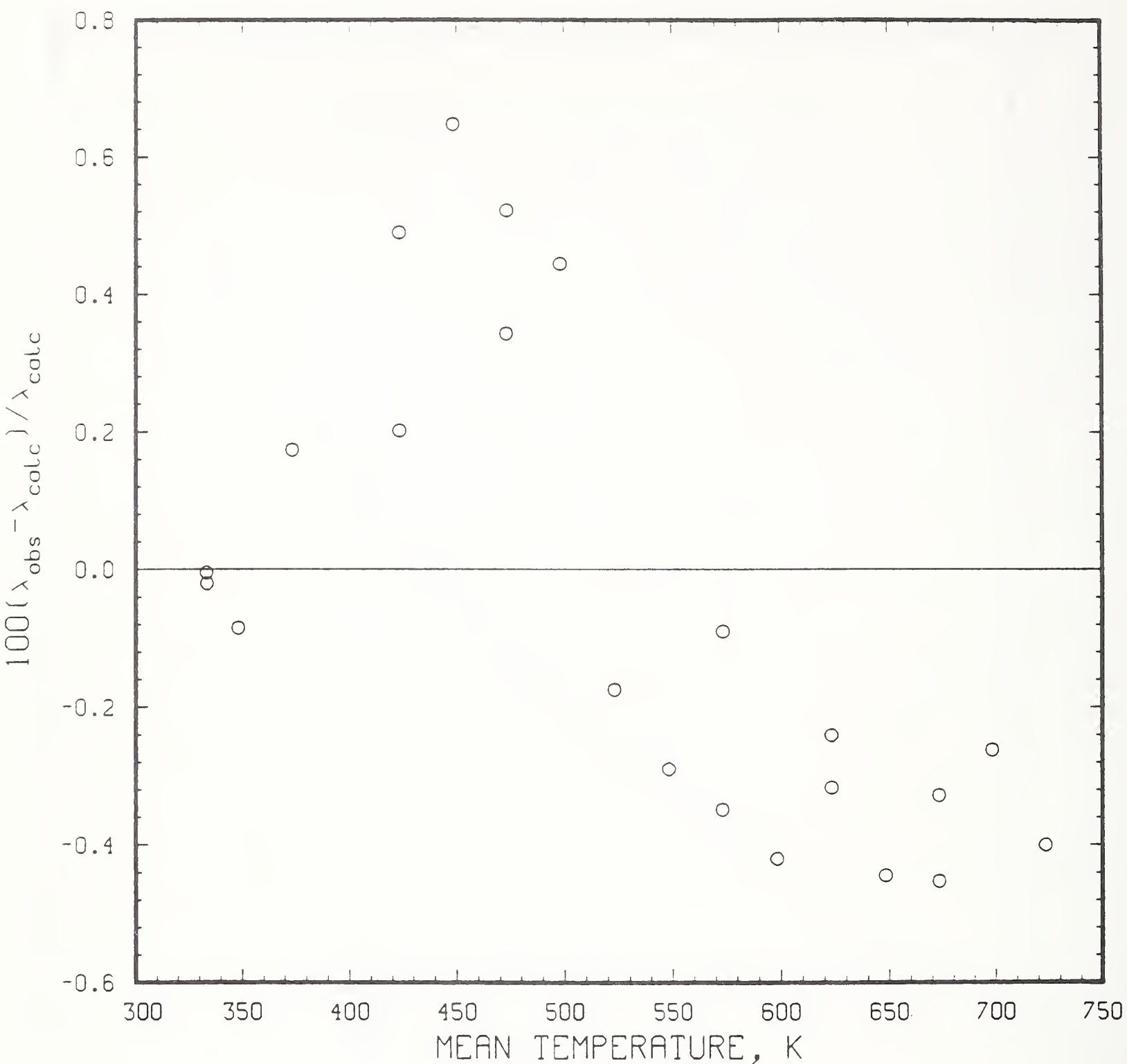


Figure 5. Relative (percent) deviations of thermal conductivity of fumed-silica insulation pair B23 from values calculated with eq (3). The standard deviation of the residuals between the points and the curve is 0.35 percent.

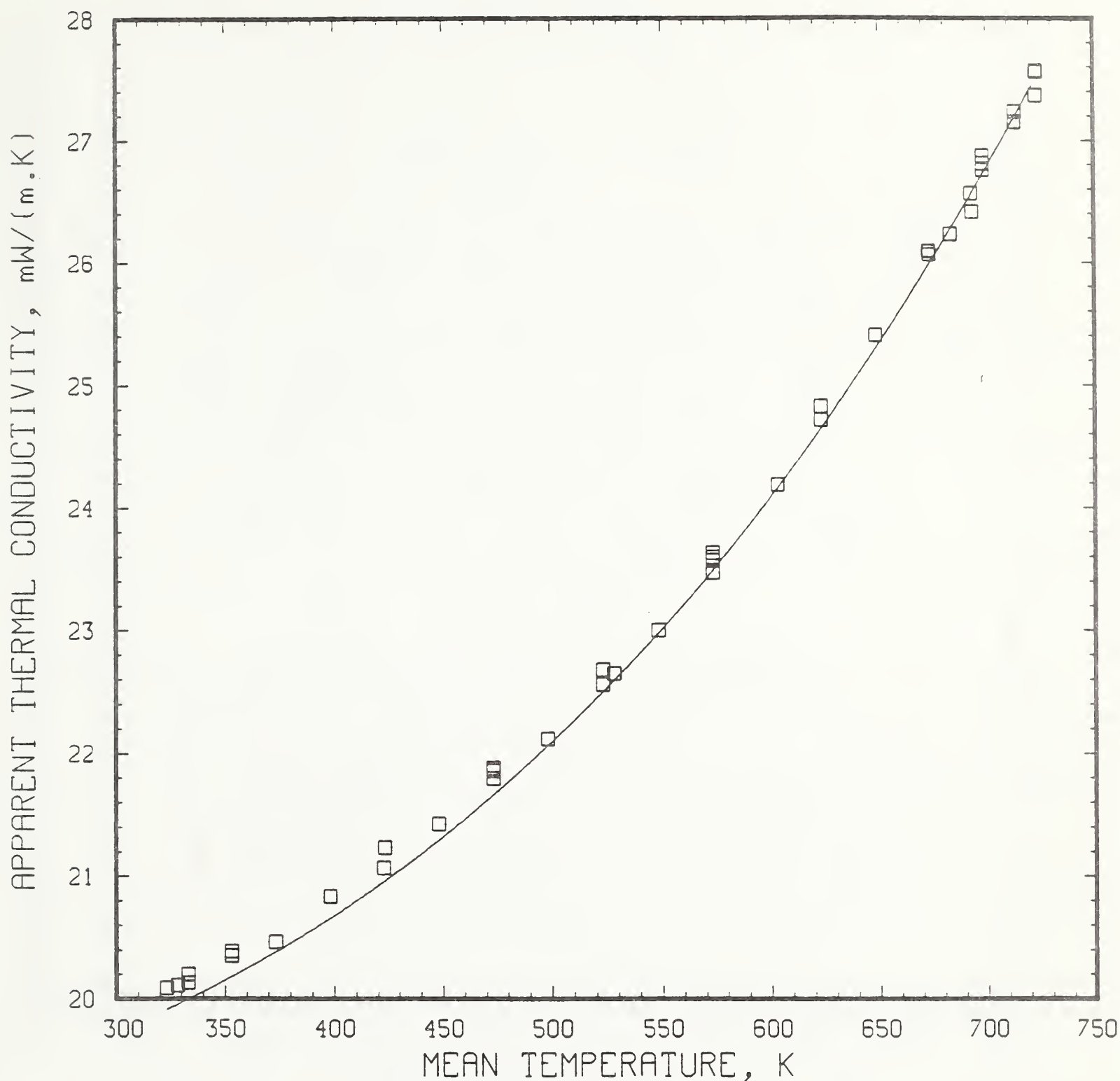


Figure 6. Apparent thermal conductivity of pair B12 of micro-porous fumed-silica insulation at mean specimen temperatures ranging from 323 to 723 K (50 to 450°C). This pair has a mean density of 304 kg/m³ (19 lbm/ft³). Conductivity was measured in air at ambient humidity and at a pressure of 83 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

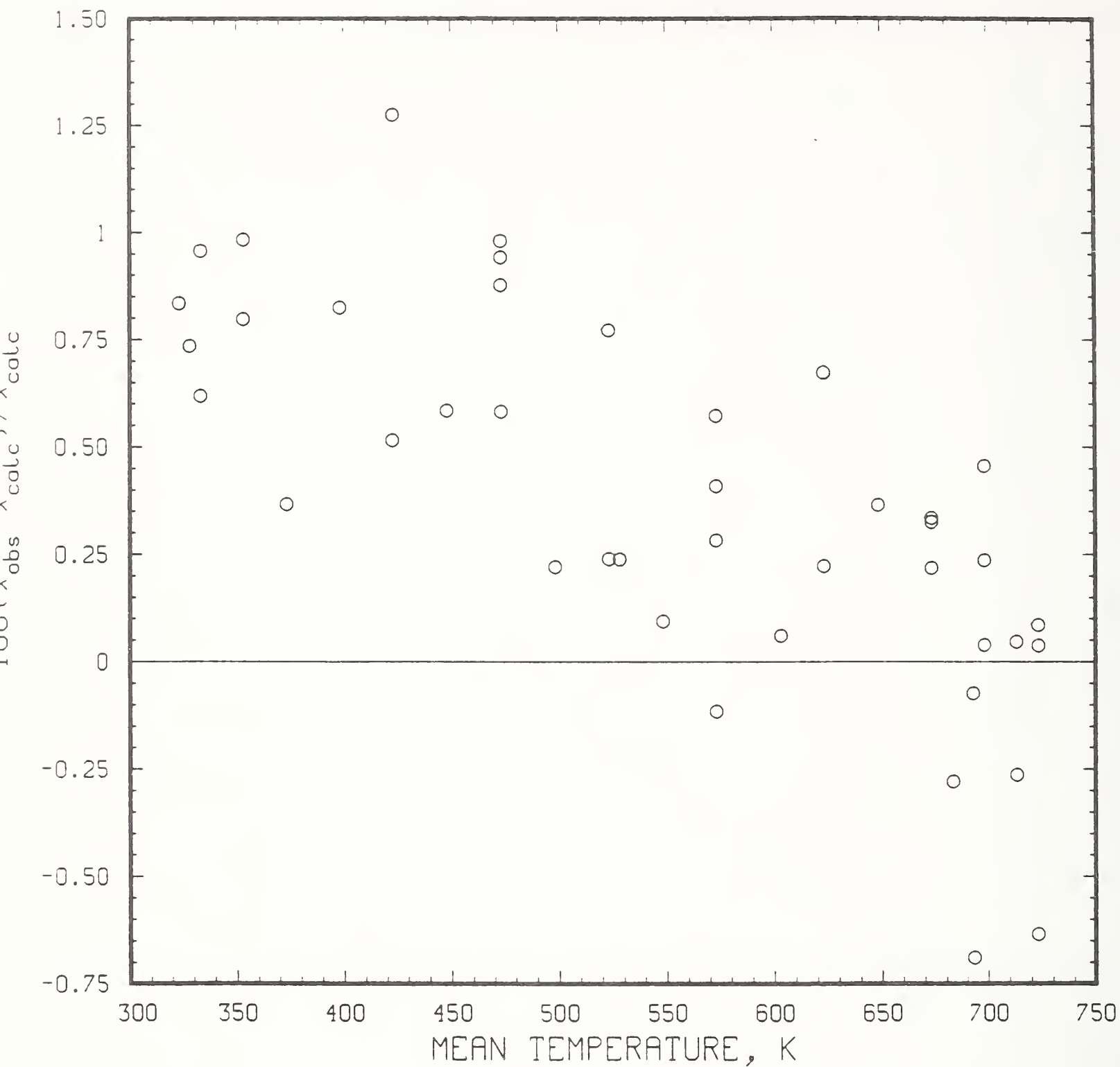


Figure 7. Relative (percent) deviations of thermal conductivity of fumed-silica insulation pair B12 from values calculated with eq (3). The standard deviation of the residuals between the points and the curve is 0.58 percent.

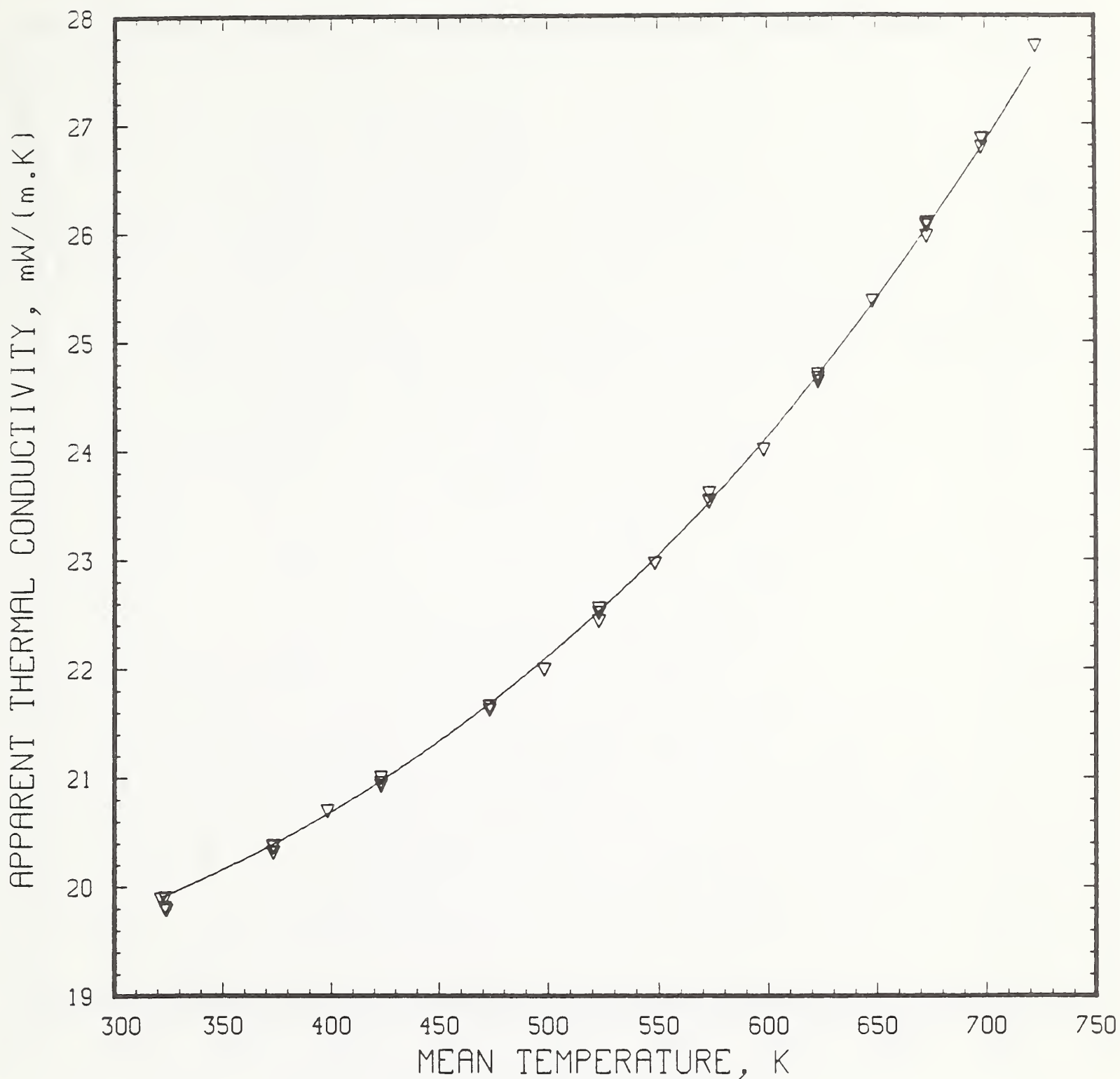


Figure 8. Apparent thermal conductivity of pair B14 of micro-porous fumed-silica insulation at mean specimen temperatures ranging from 321 to 723 K (48 to 450°C). This pair has a mean density of 301 kg/m³ (19 lbm/ft³). Conductivity was measured at ambient humidity and at a pressure of 83 kPa. The solid curve represents values of K_{calc} , obtained using eq (3).

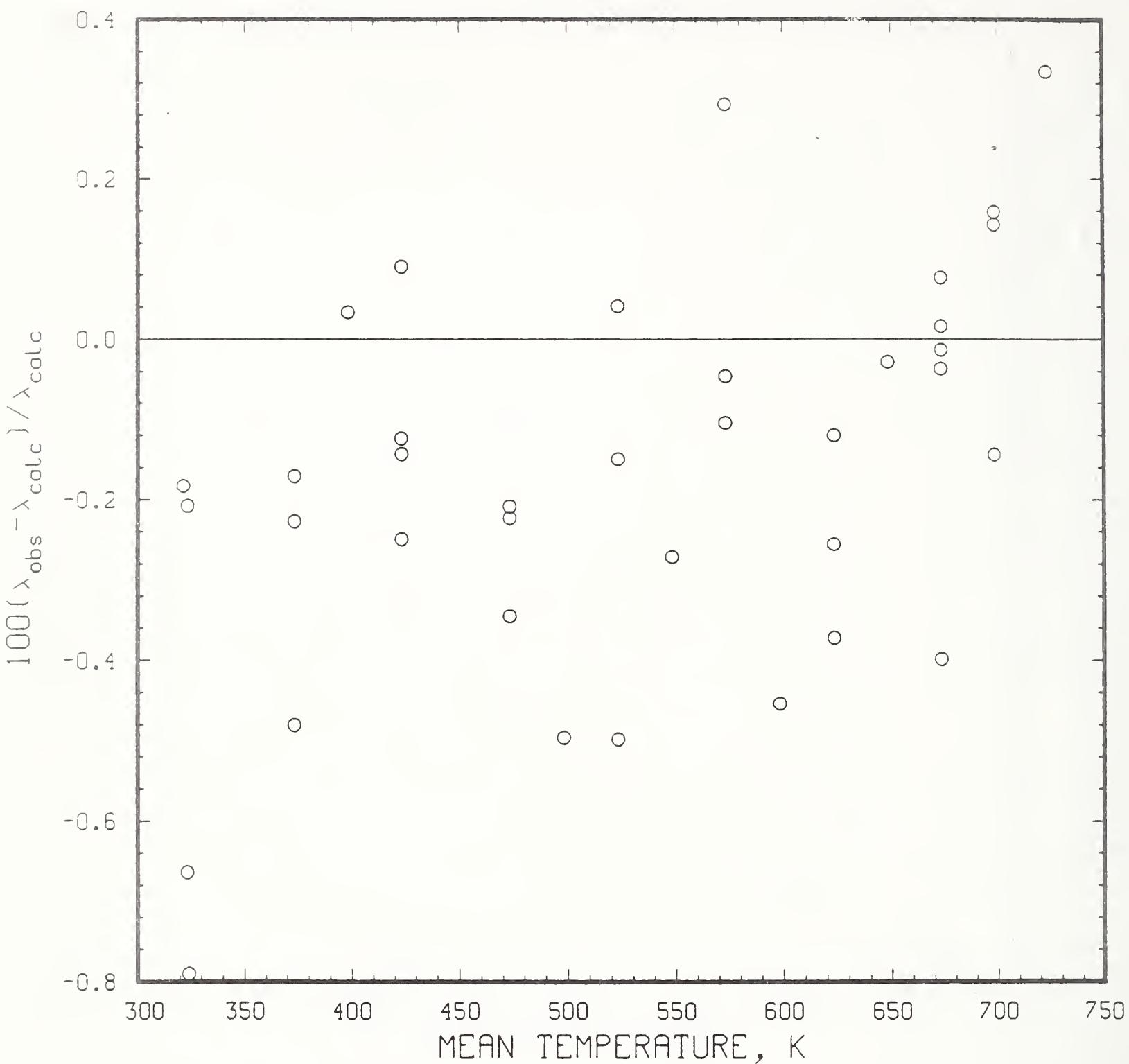


Figure 9. Relative (percent) deviations of thermal conductivity of fumed-silica insulation pair B14 from values calculated with eq (3). The standard deviation of the residuals between the points and the curve is 0.30 percent.

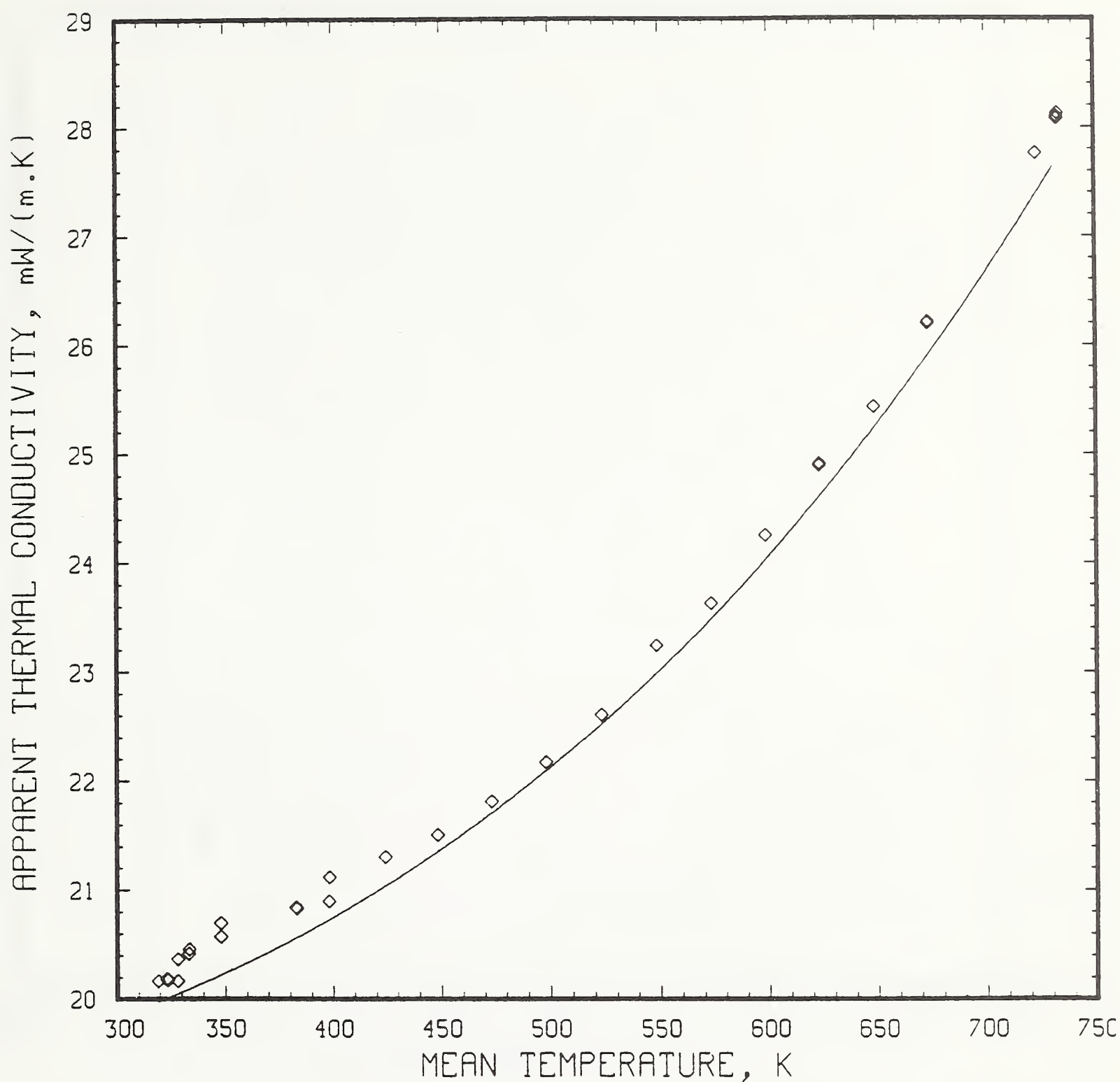


Figure 10. Apparent thermal conductivity of pair B34 of micro-porous fumed-silica insulation at mean specimen temperatures ranging from 321 to 723 K (48 to 450°C). This pair has a mean density of 315 kg/m³ (20 lbm/ft³). Conductivity was measured at ambient humidity and at a pressure of 83 kPa. The solid curve represents values of K_{calc} , obtained using eq (3).

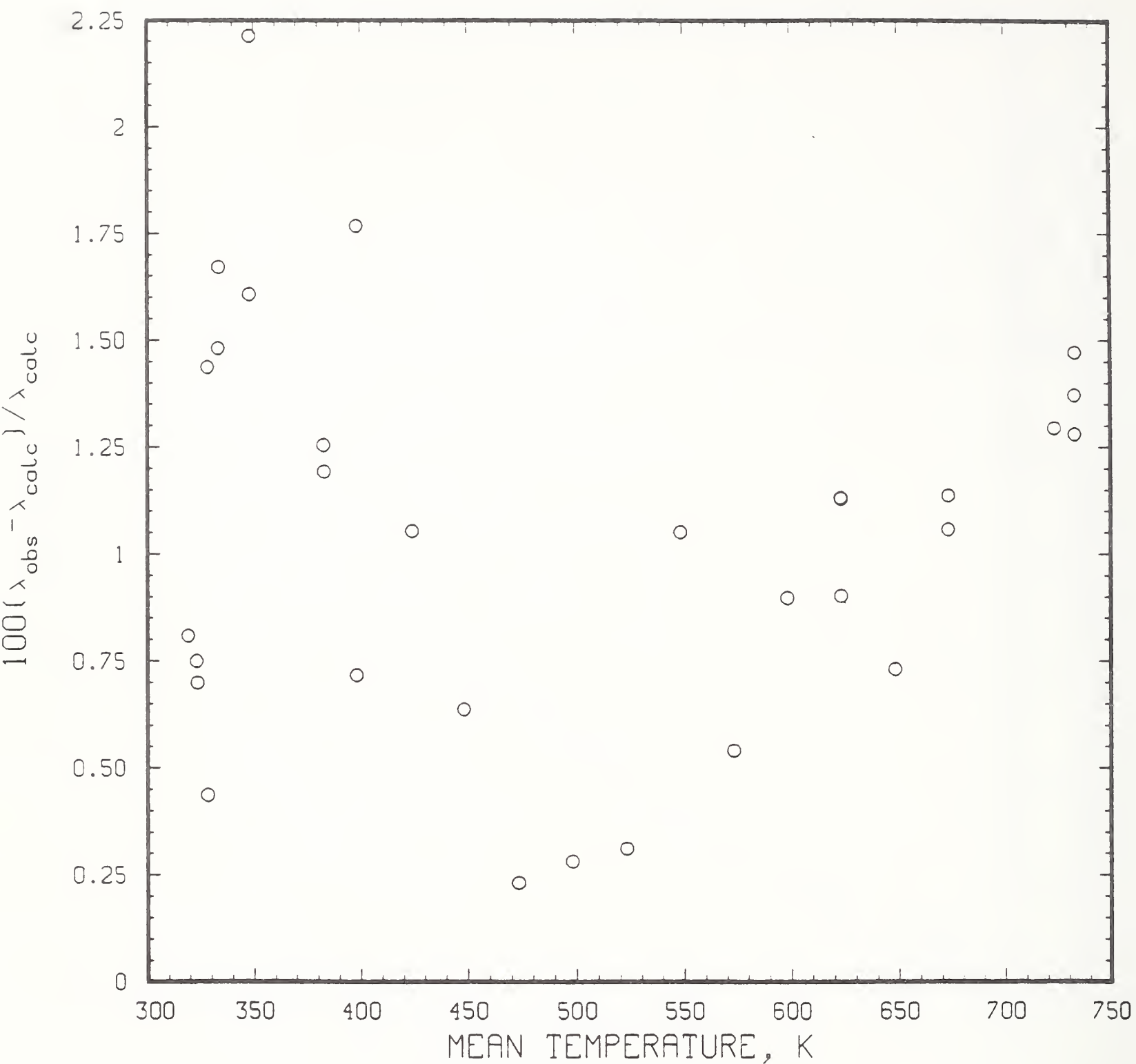


Figure 11. Relative (percent) deviations of thermal conductivity of fumed-silica insulation pair B34 from values calculated with eq (3). The standard deviation of the residuals between the points and the curve is 1.16 percent.

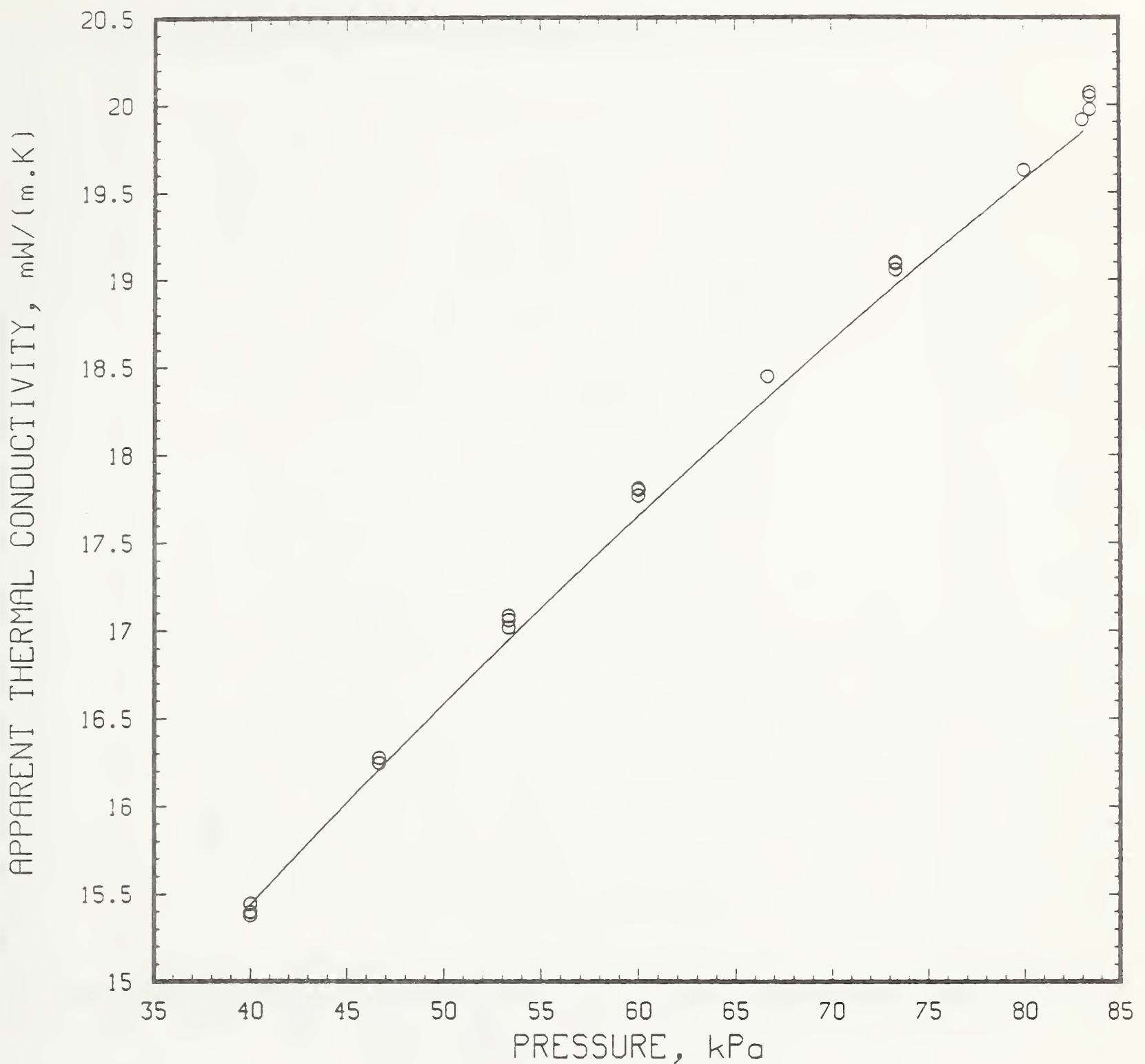


Figure 12. Apparent thermal conductivity of pair B14, density of 301 kg/m³ (19 lbm/ft³), at a mean specimen temperature of 318 K (45°C) and at pressures ranging from 40 to 83 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

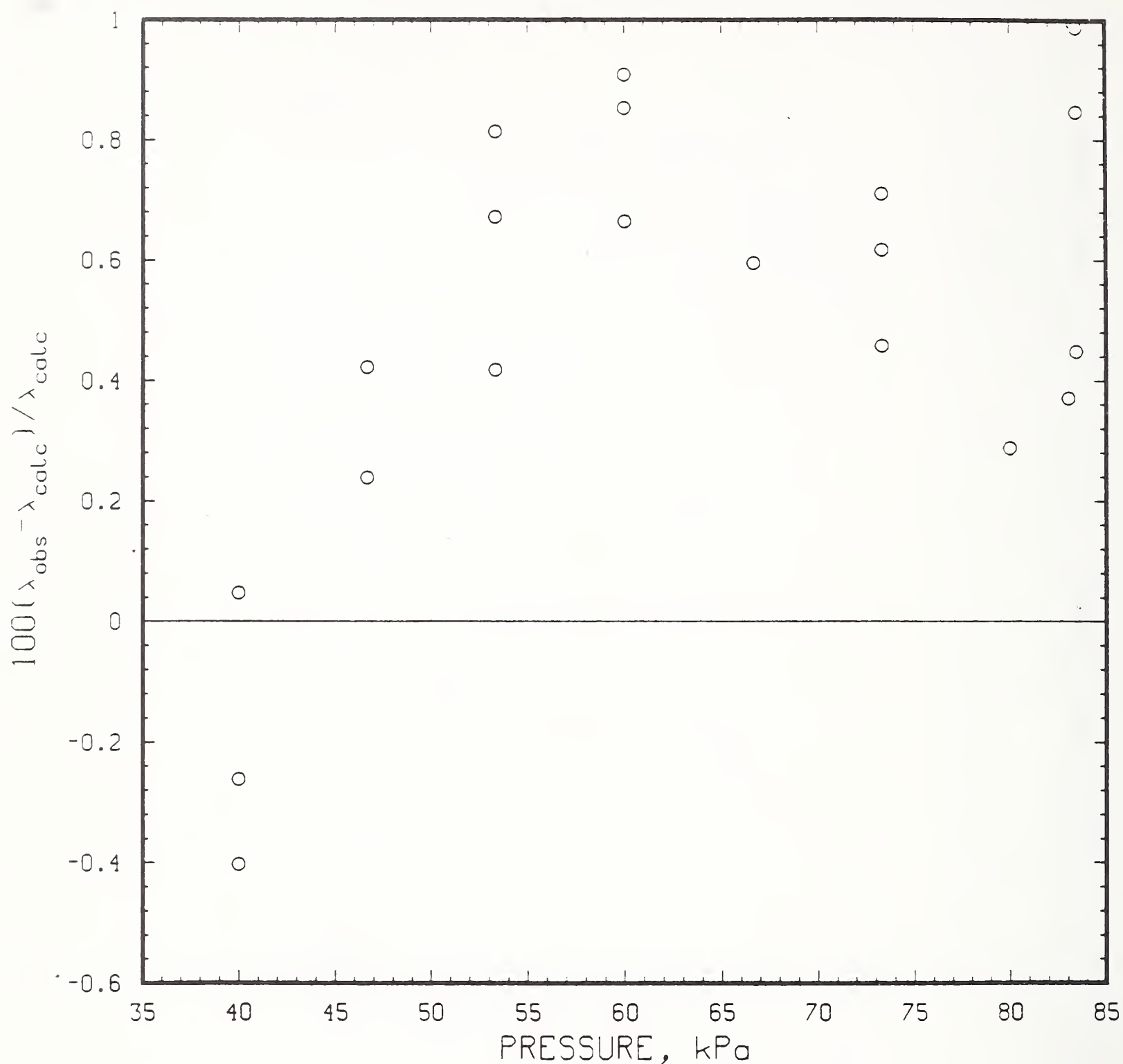


Figure 13. Relative deviation, in percent, of measured apparent thermal conductivity of fumed-silica pair B14 at 318 K, from values calculated with eq (3), over the range of pressures from 40 to 83 kPa. The standard deviation of the residuals between the points and the curve is 0.62 percent.

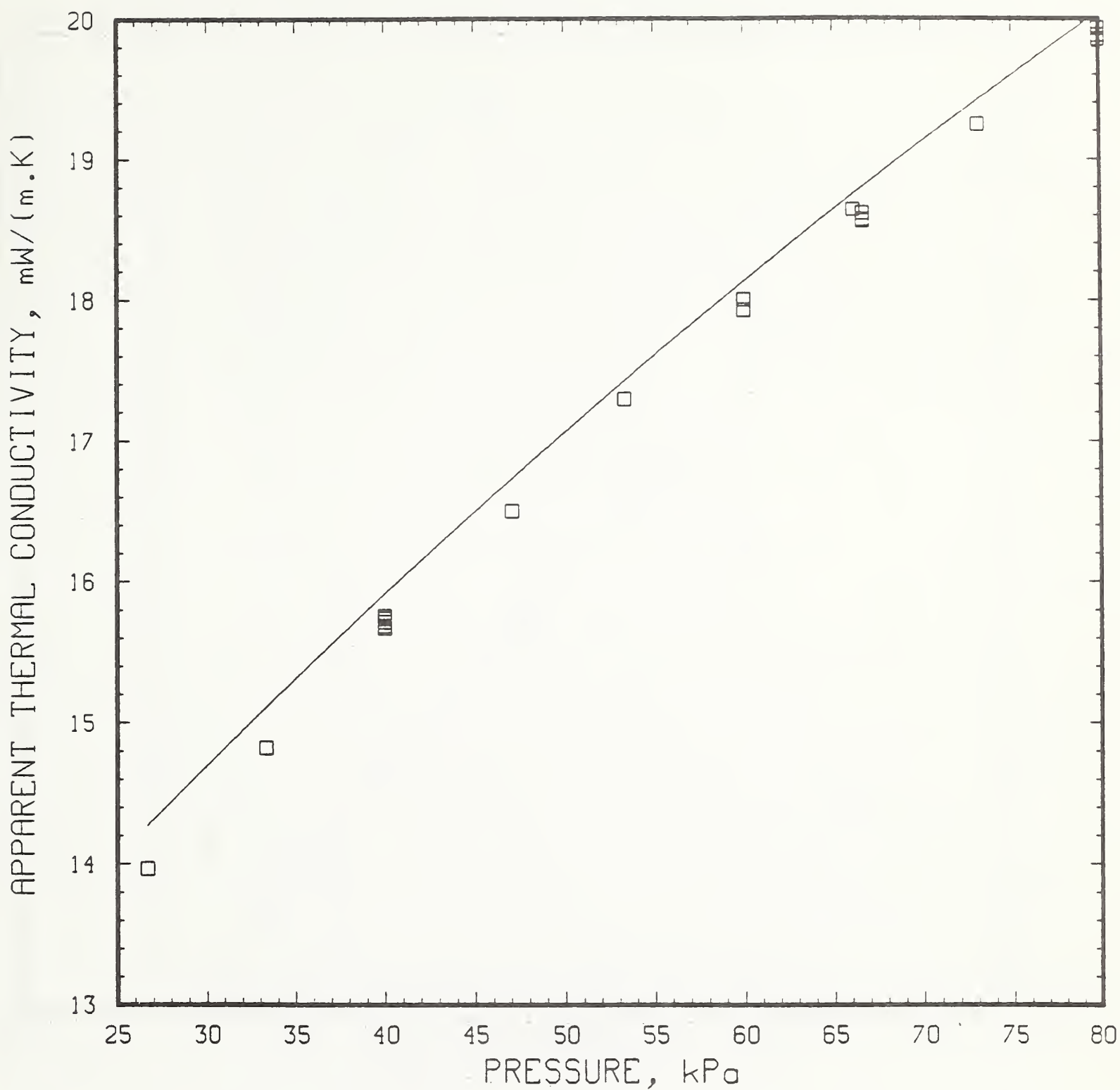


Figure 14. Apparent thermal conductivity of pair B8cd, density of 348 kg/m³ (22 lbm/ft³), at a mean specimen temperature of 318 K (45°C) and at pressures ranging from 26 to 80 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

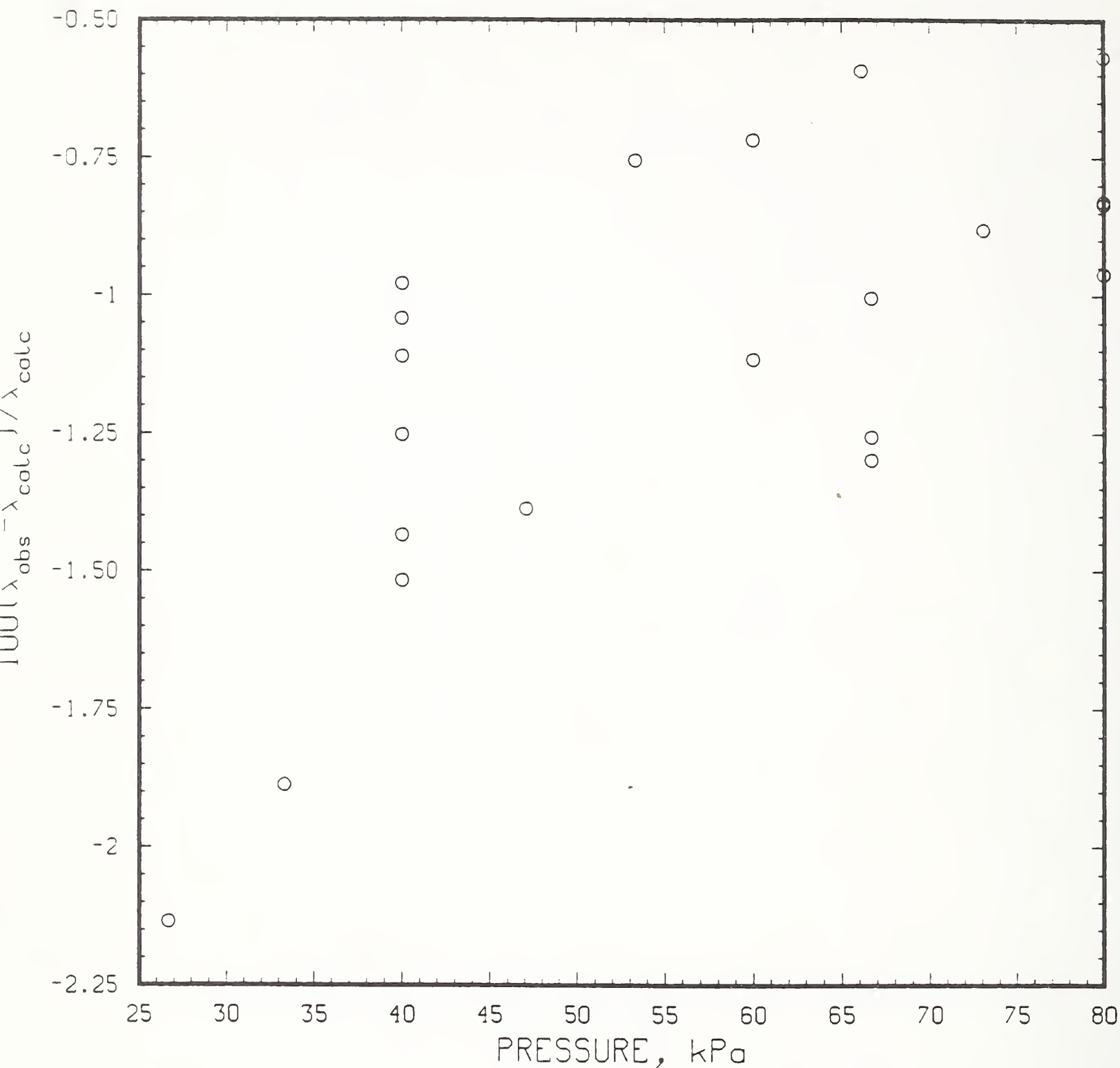


Figure 15. Relative deviation, in percent, of measured apparent thermal conductivity of fumed-silica pair B8cd at 318 K, from values calculated with eq (3), over the range of pressures from 26 to 80 kPa. The standard deviation of the residuals between the points and the curve is 1.22 percent.

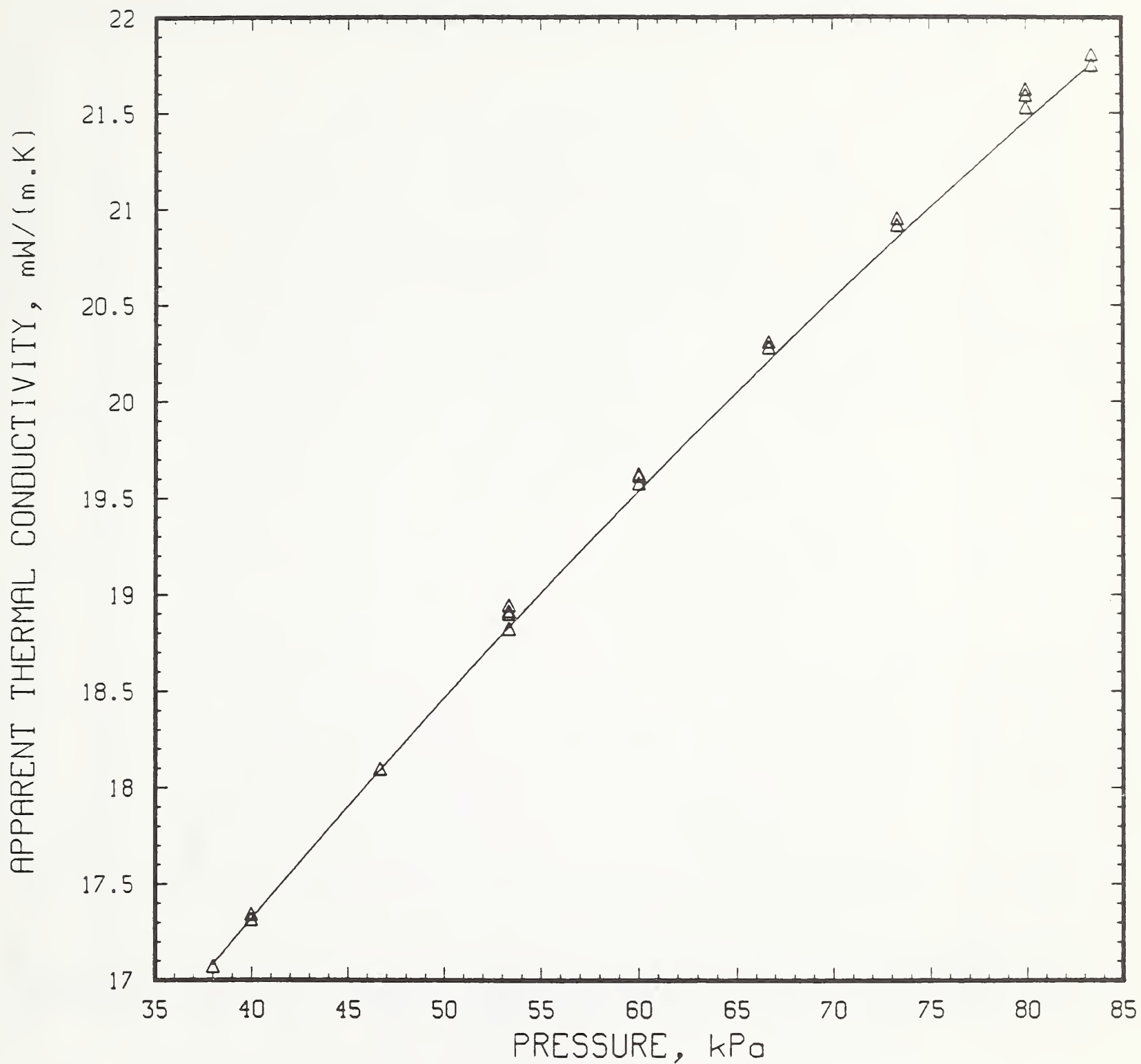


Figure 16. Apparent thermal conductivity of pair B34, density of 315 kg/m³ (20 lbm/ft³), at a mean specimen temperature of 473 K (200°C) and at pressures ranging from 37 to 83 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

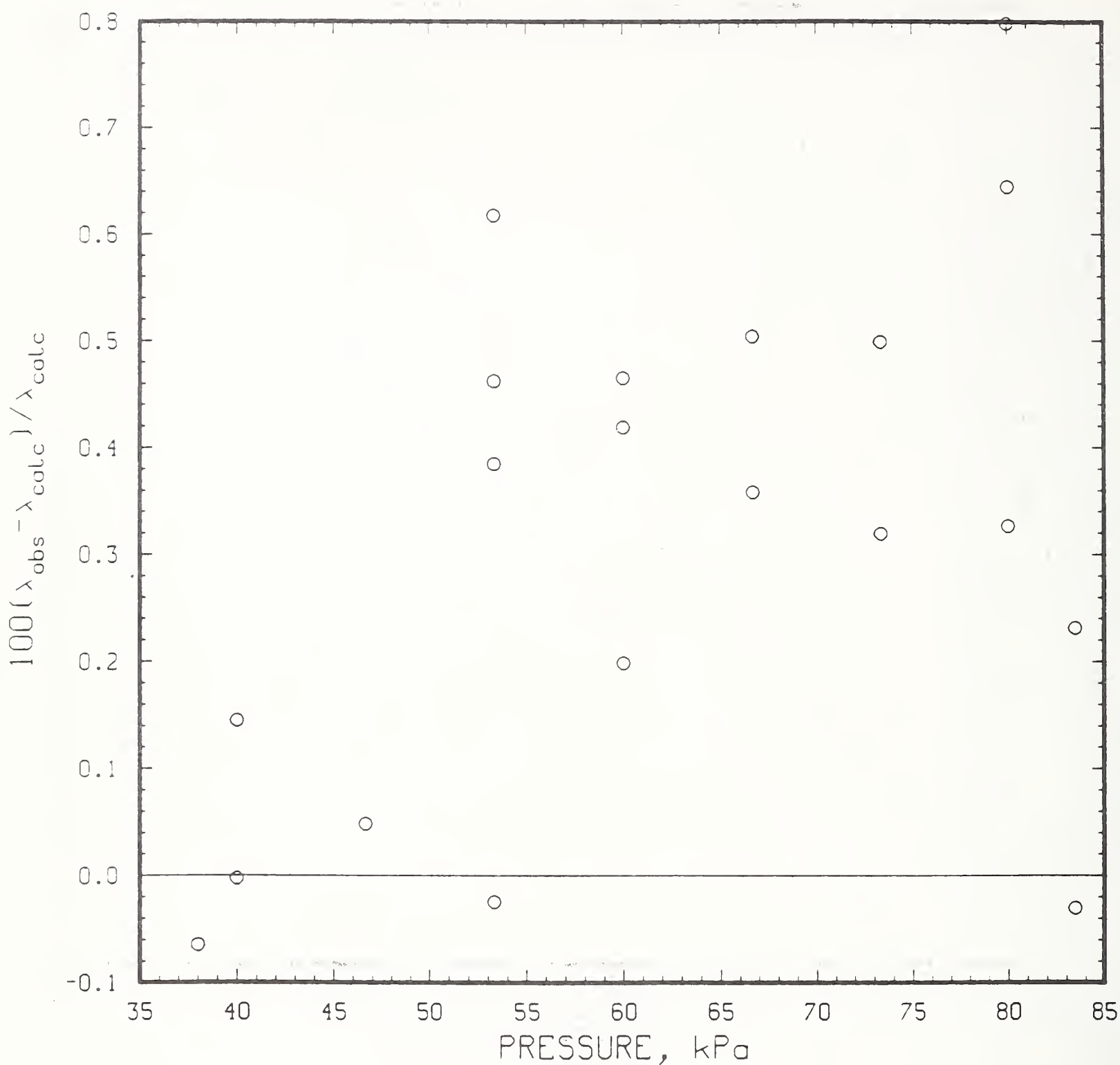


Figure 17. Relative deviation, in percent, of measured apparent thermal conductivity of fumed-silica pair B34 at 473 K, from values calculated from eq (3), over the range of pressures from 37 to 83 kPa. The standard deviation of the residuals between the points and the curve is 0.41 percent.

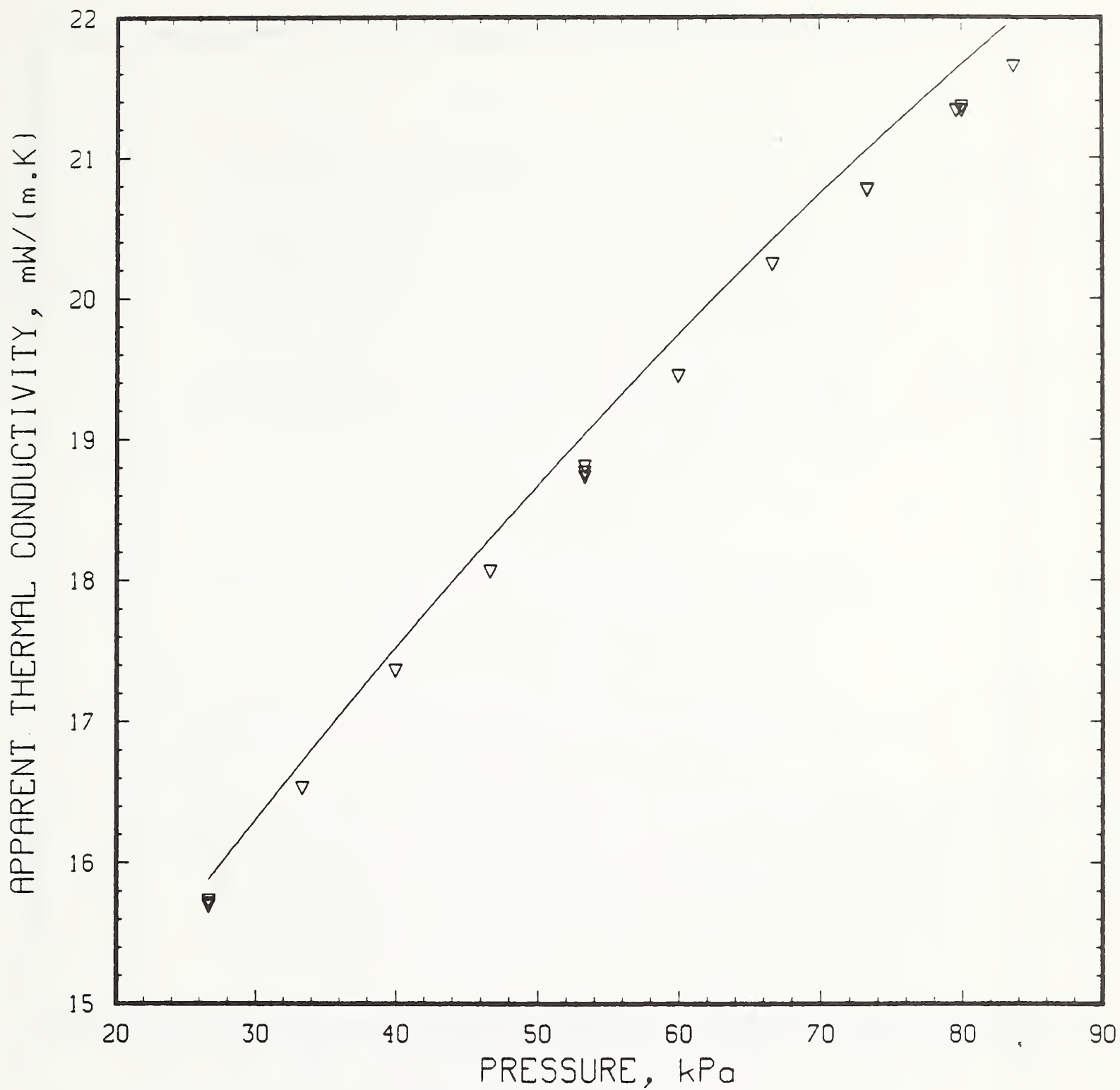


Figure 18. Apparent thermal conductivity of pair B8cd, density of 348 kg/m³ (22 lbm/ft³), at a mean specimen temperature of 473 K (200°C) and at pressures ranging from 26 to 84 kPa. The solid curve represents values of K_{calc} obtained using eq (3).

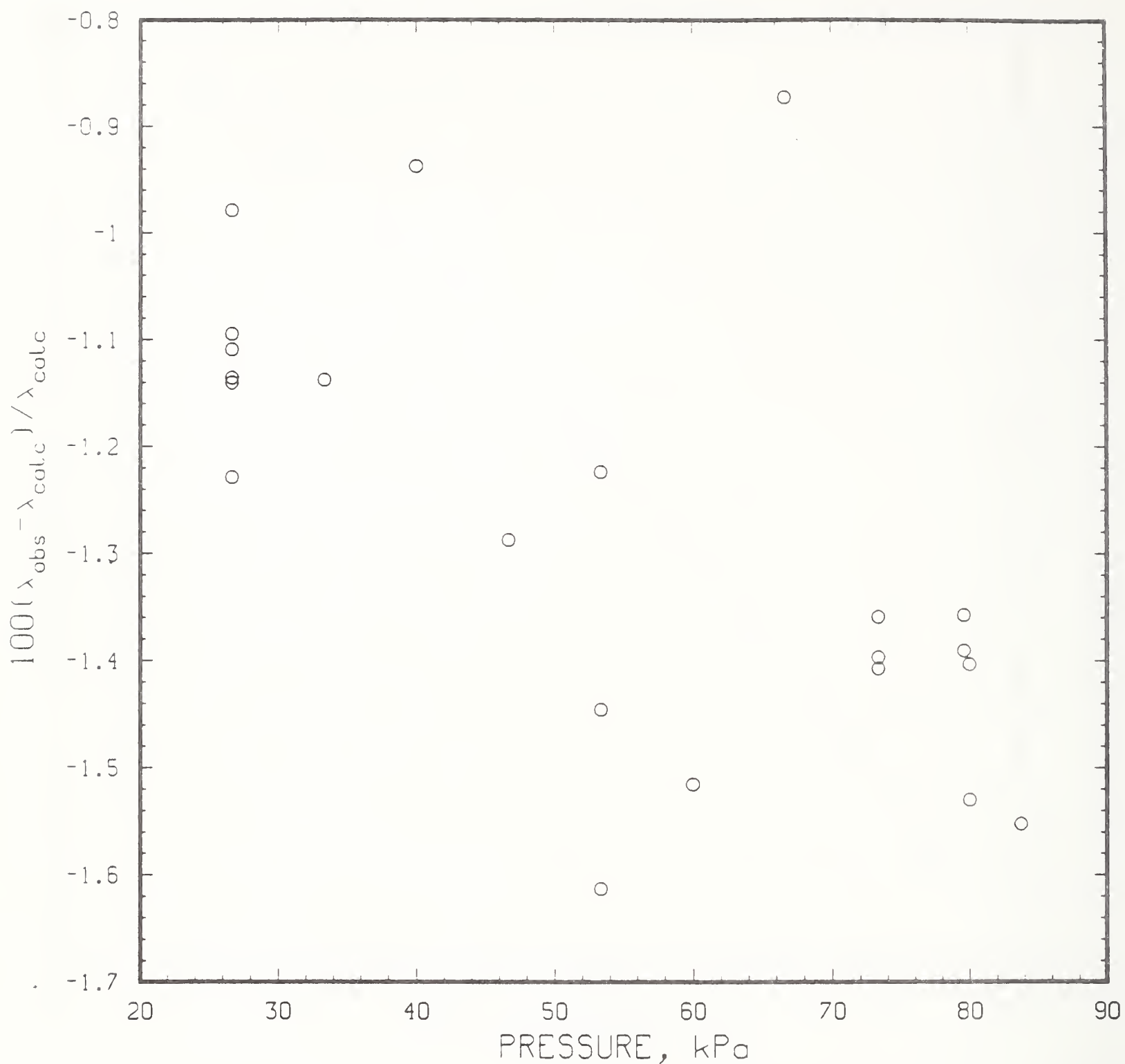


Figure 19. Relative deviation, in percent, of measured apparent thermal conductivity of fumed-silica pair B8cd at 473 K, from values calculated from eq (3), over the range of pressures from 26 to 84 kPa. The standard deviation of the residuals between the points and the curve is 1.32 percent.

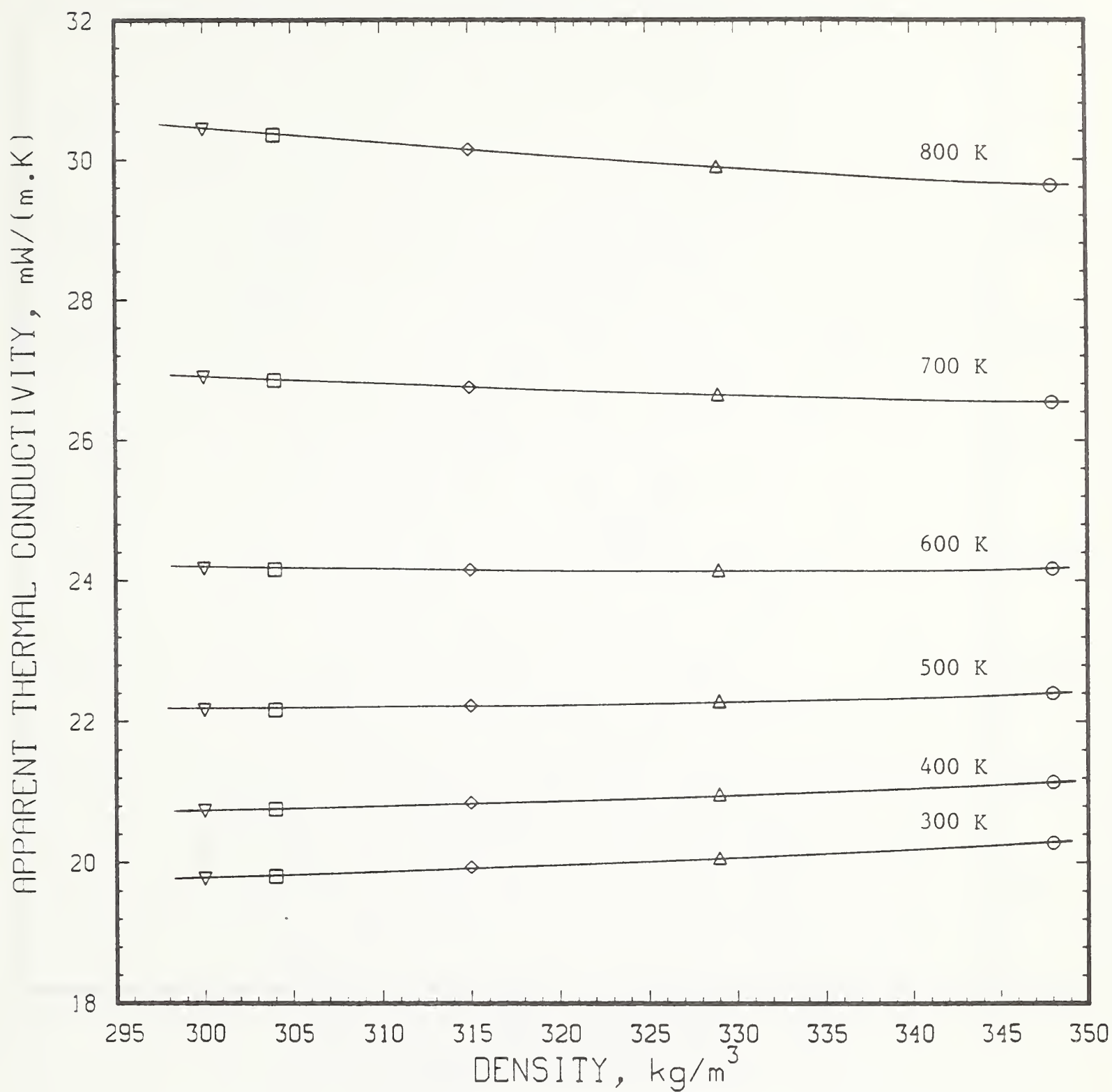


Figure 20. Apparent thermal conductivity as a function of density for five measured pairs of fumed-silica specimens, at temperatures of 300 to 800 K. Measurements were performed with an ambient air pressure of 83 kPa. Open symbols are values calculated from eq (3) for the densities of the specimen pairs measured.

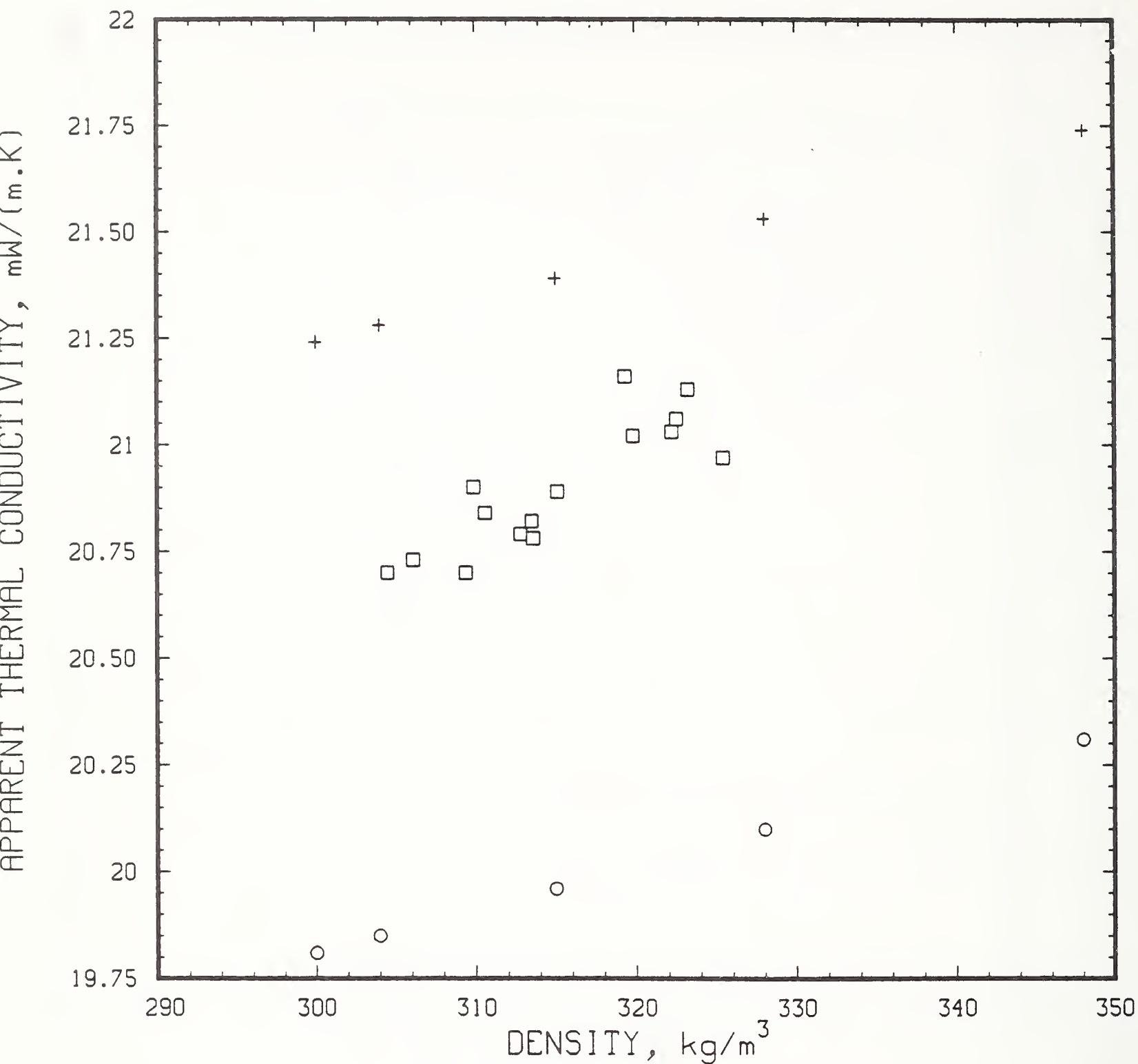


Figure 21. Dependence of apparent thermal conductivity of fumed-silica insulation on bulk density. Open squares represent measurements at 297 K and at ambient pressure of 101.3 kPa at NIST-G. Open circles represent points calculated from the least-squares fit to actual data obtained at NIST-B at 300 K and at ambient air pressure of 83.2 kPa. Plus signs are data from NIST-B extrapolated upward to a pressure of 101.3 kPa by addition of 1.43 units.

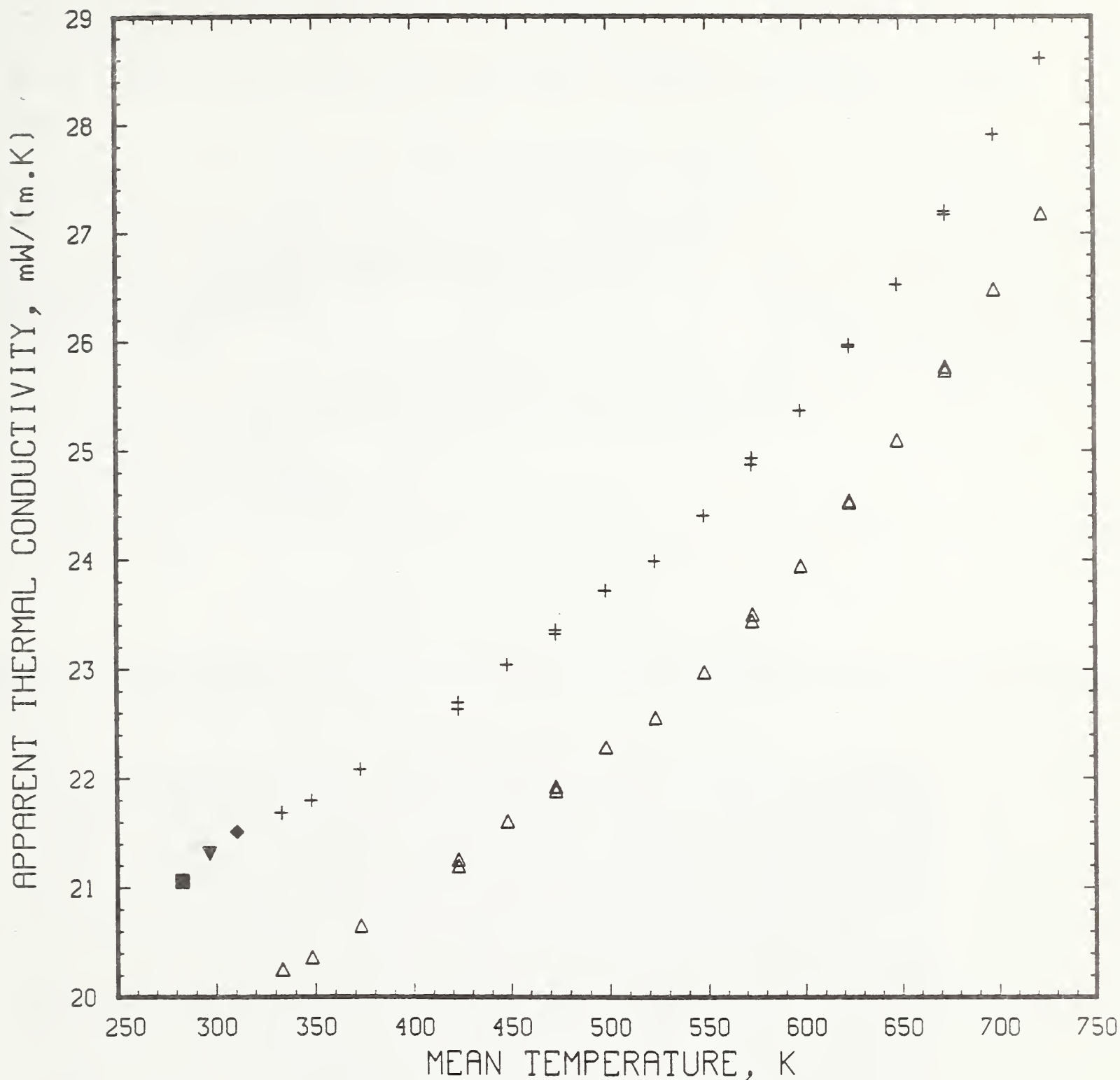


Figure 22. Dependence of apparent thermal conductivity of fumed-silica insulation on temperature, as measured at ambient air pressure of 83 kPa at NIST-B (open triangles) and at ambient pressure of 101.3 kPa at NIST-G (filled square, triangle and diamond). Plus signs are data from NIST-B extrapolated upward to a pressure of 101.3 kPa by addition of 1.43 units. Data from NIST-G are for a specimen with a density of 330 kg/m³. Data (open triangles) from NIST-B are for one with a density of 328 kg/m³ (pair B23) and are identical to data in figure 4.

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5. AUTHOR(S) DAVID R. SMITH				
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10. SUPPLEMENTARY NOTES <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.				
11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) Measurements of apparent thermal conductivity of microporous fumed-silica insulation board, already certified as a Standard Reference Material (SRM) of thermal resistance, are reported here to extend the range of certification of this material to higher temperatures and lower pressures. Apparent thermal conductivities of five different pairs of specimens ranging in mean density from 300 to 348 kg/m ³ were measured with a high-temperature guarded hot plate 25 cm in diameter. The measurements cover a range of mean specimen temperatures from 318 to 733 K (45 to 460°C), and of environmental air pressures from 26.7 to 83.5 kPa (200 to 626 Torr). Detailed analyses are given, along with intercomparisons of previously published data. Apparent thermal conductivity is correlated with temperature, pressure, and density; the correlation obtained represents all the data within a standard deviation of 0.68 percent. This microporous fumed silica (at an ambient pressure of 83 kPa and a density of 300 kg/m ³) has an apparent thermal conductivity of 19.8 mW/(m·K) at 300 K and is suitable for use as an SRM of very low conductivity from 297 to 735 K (24 to 460°C). Adsorbed moisture within this material must be driven off by prolonged heating at 110°C before its conductivity is measured. Great care in handling this material is necessary because of its fragility.				
12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) apparent thermal conductivity; density; insulation board; microporous fumed silica; pressure; Standard Reference Material; temperature; thermal insulation; thermal resistance.				
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